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Volume 3
Sampling and Analysis Plan (SAP)
Phase 1, Task 4 Field Investigation

Draft

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Prepared for
Wright-Patterson Air Force Base
2750 Air Base Wing
Office of Environmental Management

Prepared by
IT Corporation
Cincinnati, Ohio

for Environmental Management Operations
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with the U.S. Department of Energy

Environmental Management Operations
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APM	Assistant Project Manager
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
DOT	Department of Transportation
DRMO	Defense Reutilization and Marketing Office
EMO	Environmental Management Operations
EMR	Environmental Management, Restoration Branch
EPA	U.S. Environmental Protection Agency
FFS	Focused Feasibility Study
FI	Field Investigation
FID	Flame Ionization Detection
FSP	Field Sampling Plan
GC	Gas Chromatography
HSA	Hollow Stem Auger
HSC	Health and Safety Coordinator
HSP	Health and Safety Plan
ICP	Argon Plasma Emission Spectrometry
IT	International Technology Corporation
ITAS	International Technology Analytical Services
L	Liter

mg	milligram
ml	milliliter
MS	Mass Spectrophotometry
MSD	Matrix Spike Duplicates
NBS	National Bureau of Standards
NIH	National Institute of Health
OEM	Office of Environmental Management
OEPA	Ohio Environmental Protection Agency
OVA	Organic Volatile Analyzer
PID	Photo Ionization Detection
PM	Project Manager
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RI	Remedial Investigation
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SC	Site Coordinator
SOP	Standard Operating Procedures
SOW	Statement of Work
SSO	Site Safety Officer

TCL	Target Compound List
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbons
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WPAFB	Wright-Patterson Air Force Base

1.0 INTRODUCTION

In April 1990, Wright-Patterson Air Force Base (WPAFB), initiated an investigation to evaluate a potential Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) removal action to prevent, to the extent practicable, the offsite migration of contaminated ground water from WPAFB. WPAFB retained the services of the Environmental Management Operations (EMO)[a] and its principle subcontractor, International Technology Corporation (IT) to complete Phase I of the environmental investigation of ground-water contamination at WPAFB. Phase I of the investigation involves the short-term evaluation and potential design for a program to remove ground-water contamination that appears to be migrating across the western boundary of Area C, and across the northern boundary of Area B along Springfield Pike.

Primarily, Task 4 of Phase I focuses on collection of information at the Area C and Springfield Pike boundaries of WPAFB. Only these areas will be addressed in Task 4; additional field investigation efforts may be undertaken as Phase III of the investigation.

This Sampling and Analysis Plan (SAP) has been prepared to assist in completion of the Task 4 field investigation and is comprised of the Quality Assurance Project Plan (QAPP) and the Field Sampling Plan (FSP). The SAP has been developed to ensure appropriate levels of quality assurance and quality control during

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[a] EMO is operated for the U.S. Department of Energy by Battelle Memorial Institute.

sampling and sample analyses. The QAPP serves as a controlling mechanism during the field investigation to provide the quality assurance and control techniques needed so that the data collected are valid, reliable, and defensible. The FSP has been developed to specify all field activities and the manner in which they are to be executed.

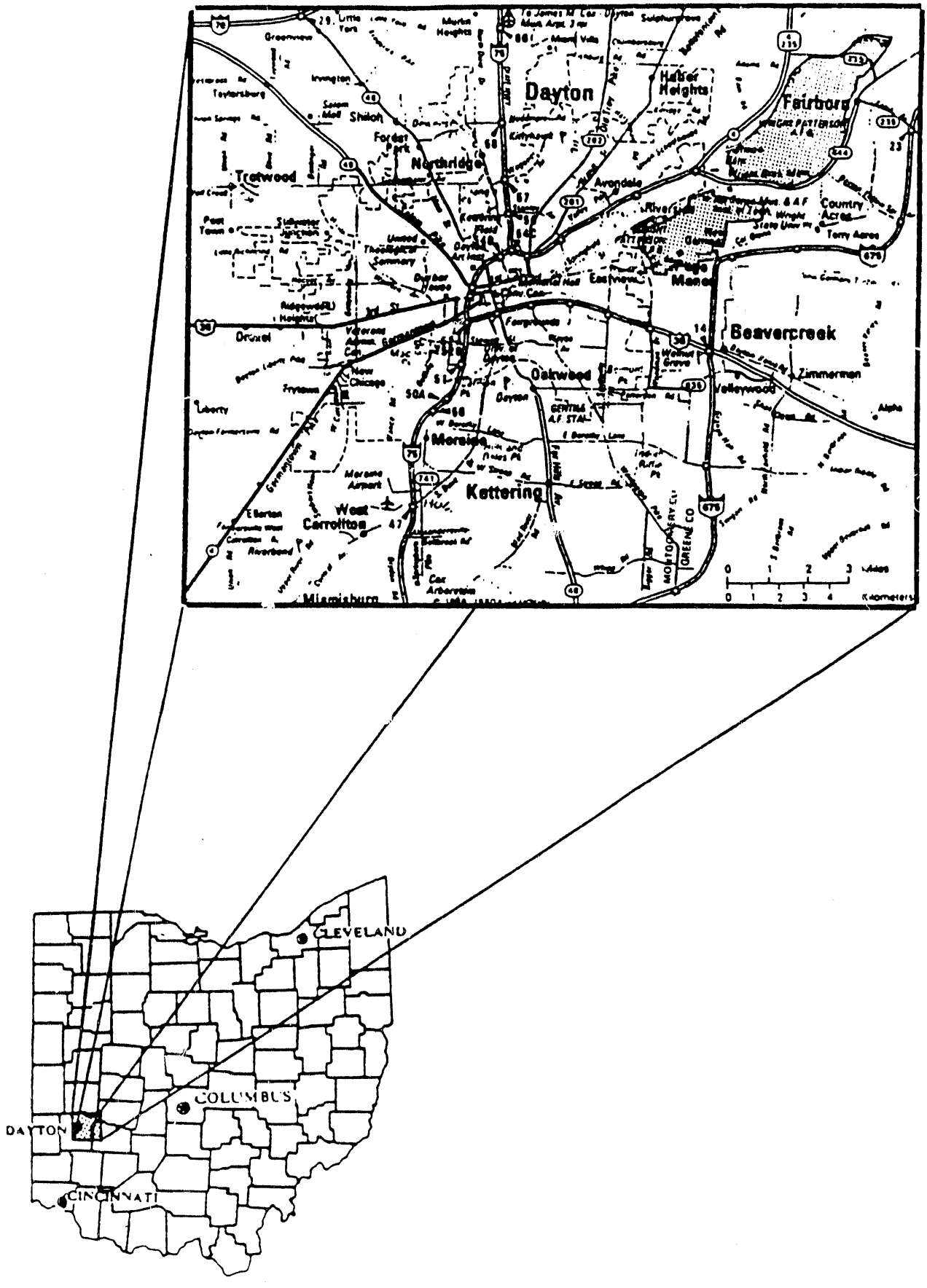
The QAPP was prepared in accordance with guidance from the U.S. Environmental Protection Agency , "Interim Guidance and Specifications for Preparing Quality Assurance Project Plans," (QAMS-005/80), and the FSP was prepared in accordance with paragraph 2.3.2.3 of the, "Guidance for Conducting Investigations and Feasibility Studies under CERCLA," (U.S. EPA/540/GT89/004). The field investigation activities associated with the FSP will be conducted in accordance with the Health and Safety Plan (HSP) submitted under separate cover.

2.0 PROJECT DESCRIPTION

WPAFB is located in southwest Ohio, between the cities of Dayton and Fairborn and occupies portions of Greene and Montgomery counties (Figure 2-1). The base is composed of two sections; Areas A and C, and Area B (Figure 2-2). Areas A and C which comprise 5,700 acres are separated from Area B by Route 444 and Conrail tracks. Area B covers about 2,800 acres and contains Wright Field, including the Air Force Museum and Air Force Institute of Technology.

Reference should be made to Sections 2 and 4 of the Volume 2, Work Plan for detailed descriptions of site conditions and the Task 4 field investigation program.

Figure 2-1 Area location map (Weston 1989)



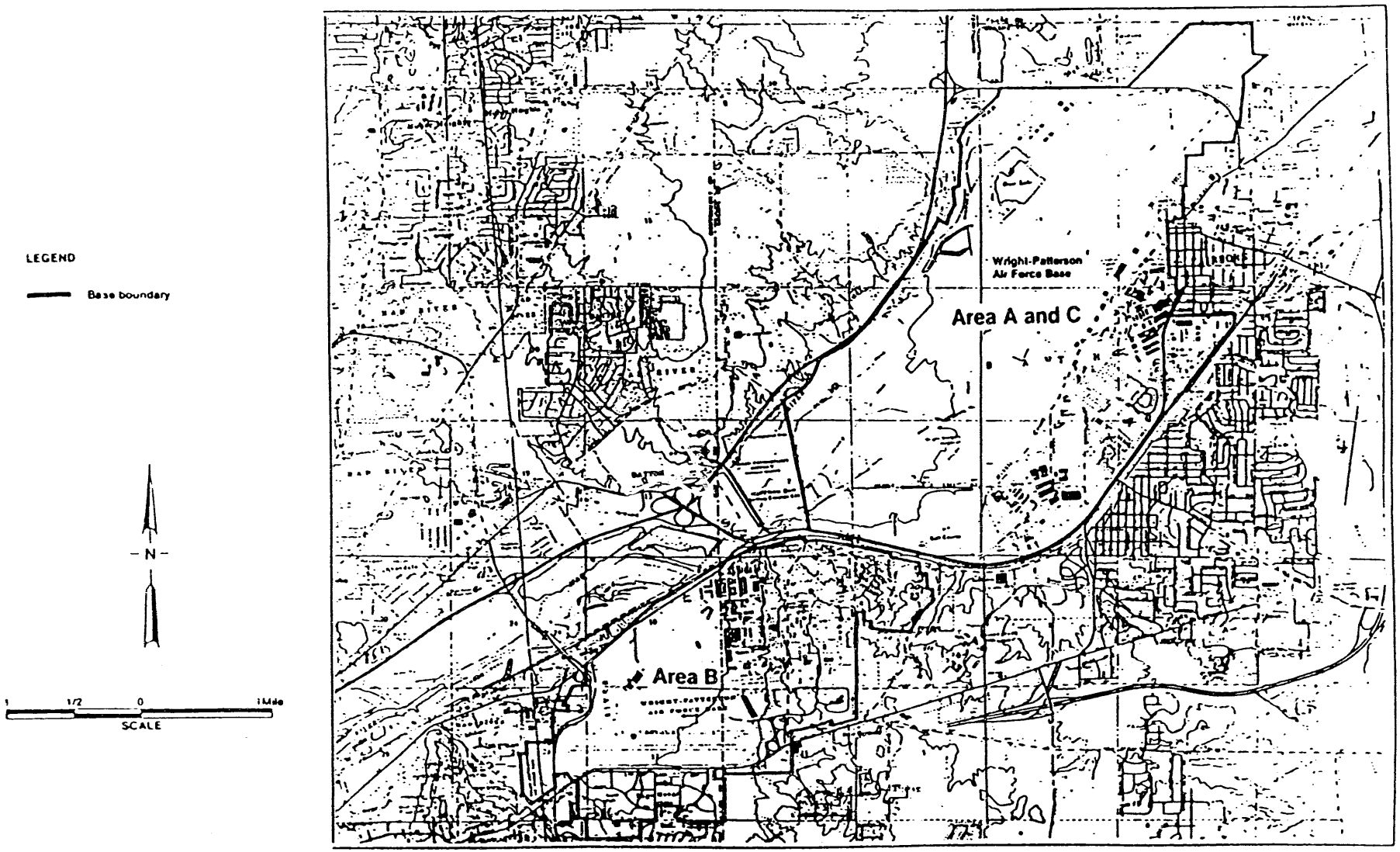


Figure 2-2 Wright-Patterson Air Force Base map (Weston 1985)

3.0 PROJECT MANAGEMENT

3.1 Project Organization and Responsibilities

WPAFB initiated an investigation to evaluate a potential CERCLA removal action to prevent, to the extent practicable, the migration of ground-water contamination in the Mad River Valley Aquifer within and across WPAFB boundaries. Environmental Management Operations (EMO) is providing overall contract management support to WPAFB. International Technology Corporation (IT) has been retained by EMO to perform the environmental investigation of ground-water contamination at WPAFB (Figure 3-1). These relationships and the key contact person in each organization are shown in Figures 3-2, 3-3 and 3-4.

3.1.1 Wright-Patterson Air Force Base

The host command for WPAFB is the 2750 Air Base Wing (ABW) which is responsible for overall execution of this project. The Office of Environmental Management (OEM) of the 2750 ABW is managing this project through its Restoration Branch (EMR). The WPAFB organization and involved personnel are shown in Figure 3-2.

Overall coordination of this project will be provided by Mr. Ronald Lester, Chief of the Restoration Branch of the Office of Environmental Management at WPAFB. He is the Base Point of Contact for those regulatory organizations involved in this project as shown in Figure 3-1. Mr. Gary Selby is the restoration project manager for this investigation.

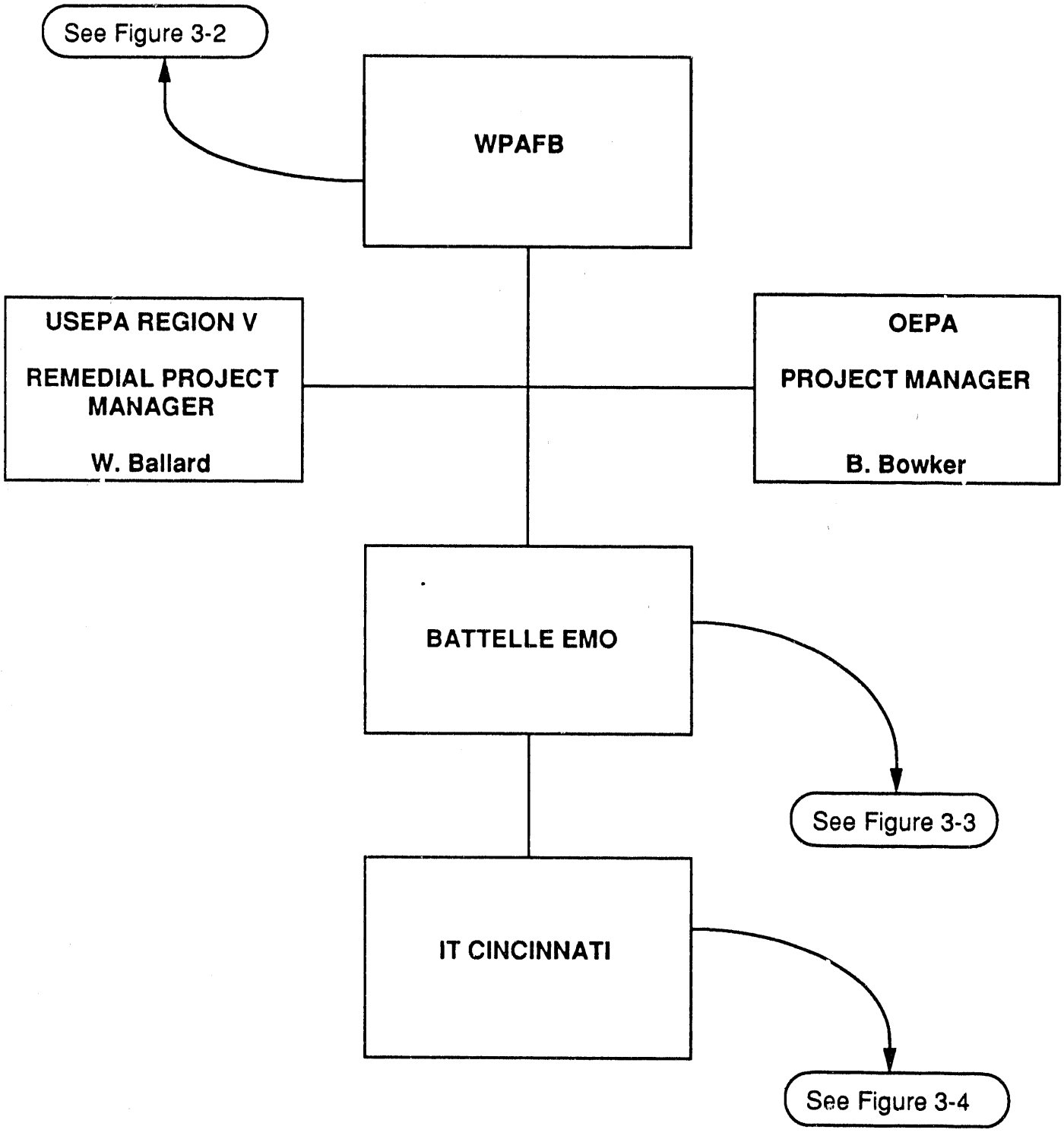


Figure 3-1. General Organization Chart.

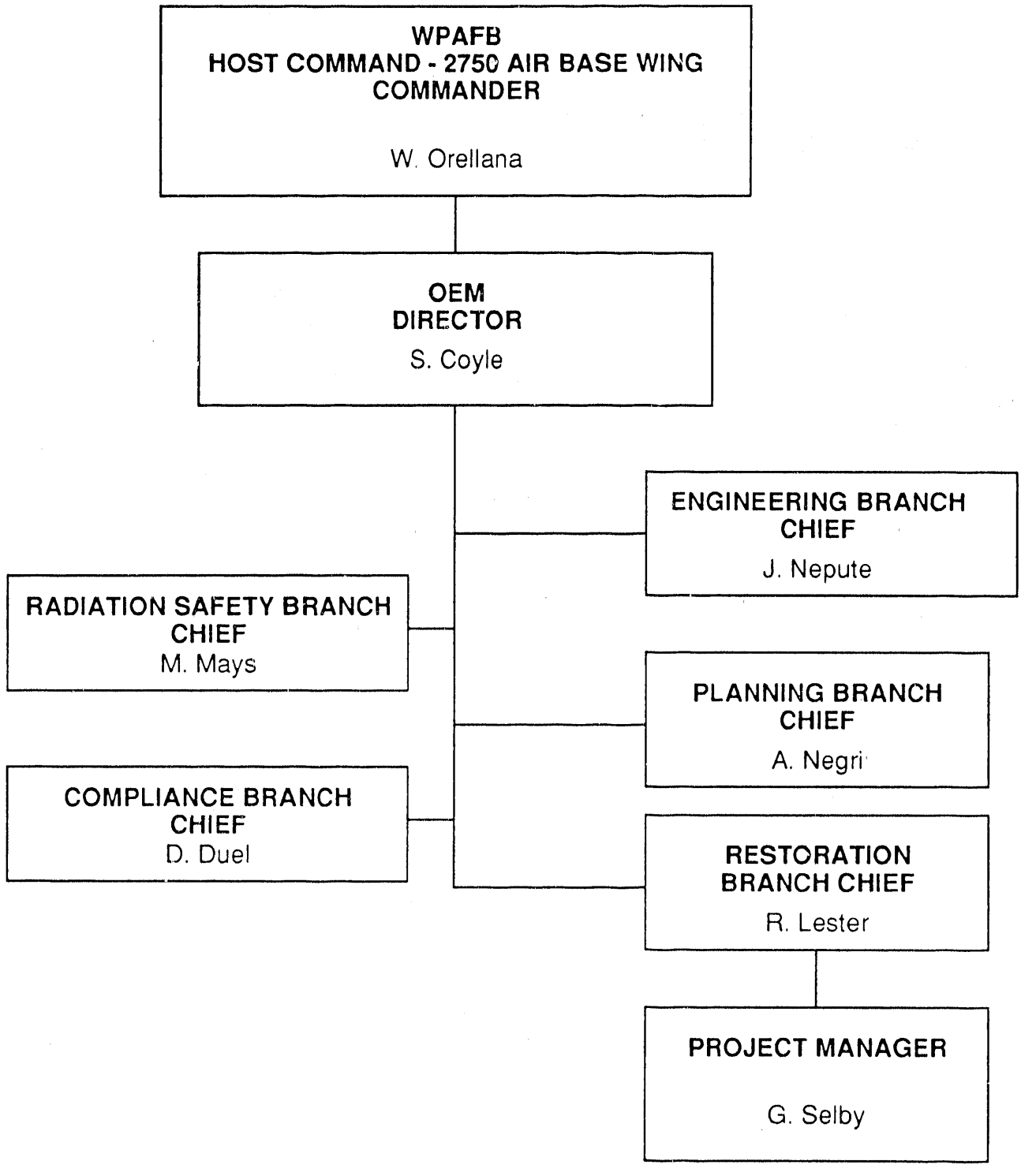


Figure 3-2. WPAFB Organization Chart.

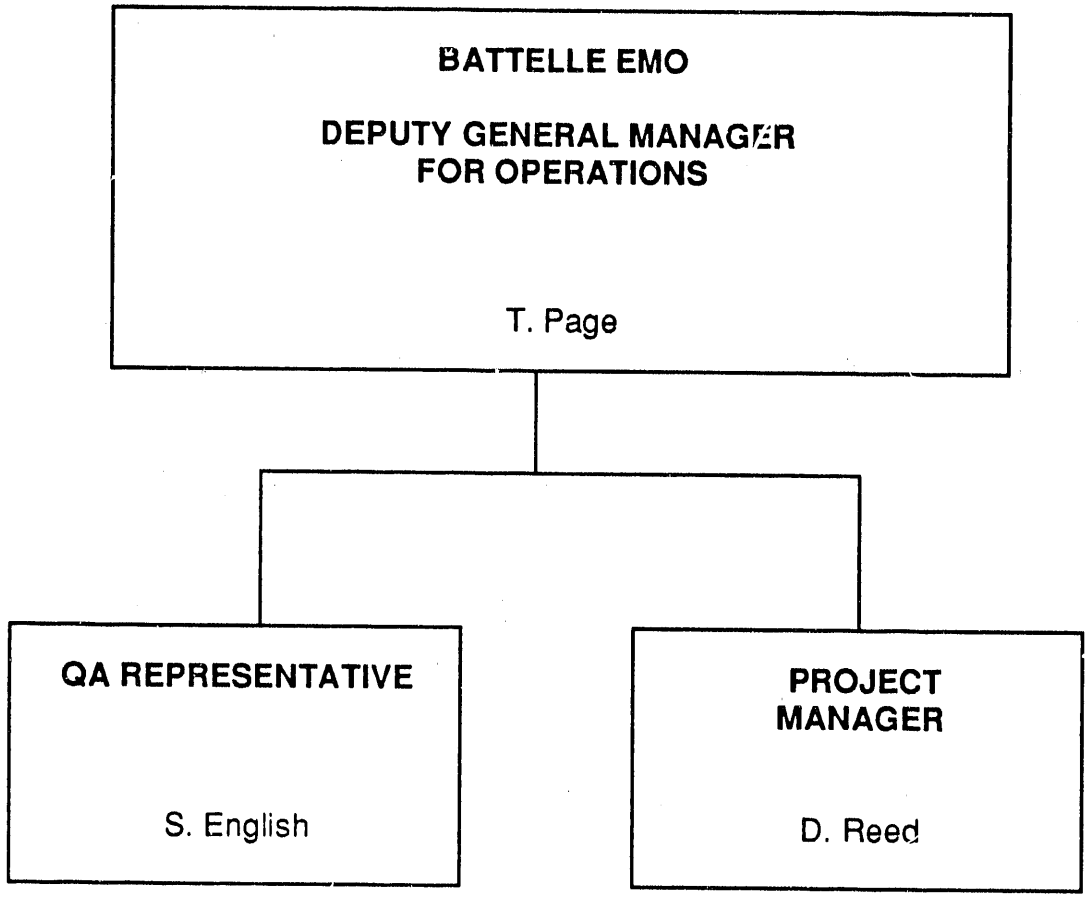


Figure 3-3. Battelle EMO Organization Chart.

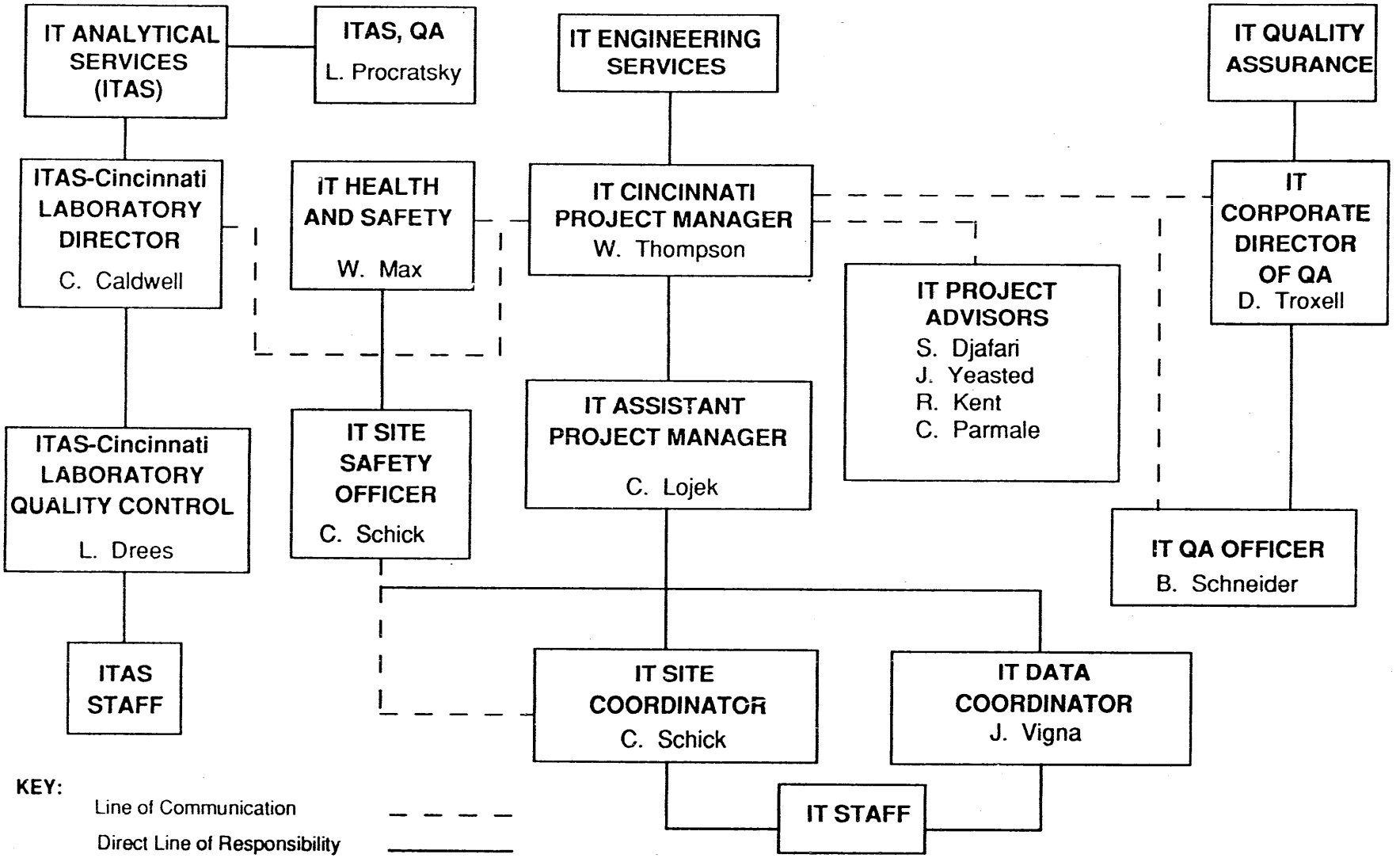


Figure 3-4. IT Organization Chart.

3.1.2 Environmental Management Operations

EMO is providing contract oversight for this project to WPAFB (Figure 3-3). Dr. Tom Page, EMO Deputy General Manager for Operations, has overall responsibility for the management of the WPAFB Project. Ms. Sandy English, the EMO QA Representative, is responsible for assuring that QA requirements for the WPAFB are met. The EMO Project Manager is Mr. Denny Reed. Mr. Reed is responsible for managing a contractor to perform this investigation. IT is the contractor retained to perform this investigation.

3.1.3 International Technology Corporation

3.1.3.1 Project Organization and Responsibilities

The organization and functions within International Technology Corporation (IT) assigned to perform this project are described below and shown in Figure 3-4.

3.1.3.1.1 Project Manager (PM)

The Project Manager, Mr. Bill Thompson, will be the prime point of contact with EMO and WPAFB and will have day-to-day responsibility for technical, financial, and scheduling matters. Mr. Thompson will also serve as the interface with the Project Advisors. Other duties, as necessary, will include:

- Procurement, along with administrative personnel, and supervision of subcontractor services.
- Assignment of duties to the project staff and orientation of the staff to the needs and requirements of the project.
- Coordination of the efforts of the project advisors.
- Approval of IT project-specific procedures and internally prepared plans, drawings, and reports.

- Dissemination of project-related information from EMO and WPAFB and others.

- Liaison between the project staff and other internal groups, such as QA, Health and Safety, and the laboratory.

- "Collection point" for project staff reporting of nonconformances and changes in project documents and activities.

- Determination of the effect of the nonconformances and changes on the project and the appropriateness for reporting such items to EMO and WPAFB and provision of appropriate documentation for any reporting.

- Notification of the project and QA groups of the project nonconformances and changes.

- Review of project documents.

3.1.3.1.2 Assistant Project Manager (APM)

The responsibilities of the Assistant Project Manager, Ms. Carole Lojek, are to perform the tasks of the PM in the PMs absence, and act as a backup to the Site Coordinator and Data coordinator. Ms. Lojek will be a principal report writer and reviewer for all project related documents.

3.1.3.1.3 Project Site Coordinator (SC)

The Project Site Coordinator (SC), Mr. Charles Schick, will be responsible for coordination all site activities with the PM, base personnel, IT Analytical Services (ITAS), and on-site subcontractors. Mr. Schick will be responsible for completing the work in accordance with this plan and notifying the PM of any changes to the plan that may be required. The duties will include:

- Providing direction and supervision to the drilling contractor during the drilling of soil test borings and monitoring well installation.

- Insuring that appropriate field logs are maintained for project activities.
- Supervising the collection of all samples and providing for their proper handling and shipping.
- Monitoring all drilling, well installation and sampling operations to provide that the drilling contractor and sampling team members adhere to the QA provisions of the plan.
- Processing and evaluating the results of the chemical analysis for the samples prior to preparation of the Field Investigation Report.

3.1.3.1.4 Data Coordinator

The Data Coordinator, Mr. John Vigna, is stationed at an IT office equipped with complete data management services, i.e., central files, word processing, duplication and drafting. Responsibilities include:

- Receipt and acknowledgement of all data from remote activity centers, i.e., lab and field.
- Processing of data for analysis and report inclusion.
- Quality control associated with data processing.
- Coordination of report preparation.

3.1.3.1.5 Project Advisors

The Project Advisors, Mr. Sirius Djafari, Mr. Joe Yeasted, Mr. Bob Kent, and Mr. Chuck Parmale, will provide the Project Manager with input to project plans, procedures, and conclusions. These senior individuals have a broad range of experience with characterization of hazardous waste sites, contaminant transport, and development and implementation of remedial measures.

3.1.3.1.6 Laboratory Director

Responsibilities of the ITAS-Cincinnati Laboratory Director, Craig Caldwell, shall include:

- General supervision of the laboratory
- Collaboration with the project group in establishing sampling and testing programs
- Schedule and execution of testing programs
- Liaison between the laboratory staff and other personnel
- "Collection point" for laboratory staff reporting of nonconformances and changes in laboratory activities
- Notification of the laboratory and QA groups of specific laboratory nonconformances and changes
- Maintenance of laboratory data and checkprints
- Release of testing data and results
- Calibration of equipment
- Storage of samples.

3.1.3.1.7 Laboratory Quality Control

Quality Control Officer, Ms. Lauren Drees, will be responsible for QC at the ITAS-Cincinnati Laboratory.

3.1.3.1.8 Corporate Director of QA

The responsibilities of the Corporate Director of QA, Mr. Dave Troxell, include:

- Administration of the corporate QA program
- Review and approval of this plan
- Supervision of QA activities

- Notification of personnel of nonconformances and changes in QA procedures
- Determination of audit schedule.

The Corporate Director of QA reports directly to the President of IT. Accordingly, Mr. Troxell may take actions independent the Corporate Director of QA may take actions independent of the project group, if required for compliance with the project QA/QC Program.

3.1.3.1.9 QA Officer

The QA Officer, Mr. Barry Schneider, is responsible for the development of this plan and the day-to-day control of project QA/QC activities. Mr. Schneider will provide the necessary guidance to the project and laboratory staffs on quality-related matters and will perform the project audits. Mr. Schneider has the authority and freedom to identify quality problems; initiate, recommend, or provide corrective actions; and verify the implementation of the corrective actions.

The Laboratory Quality Control Coordinator (QCC) is responsible for the implementation of this plan in the laboratory. The QCC is immediately informed of any nonconformances, assists in the determination of appropriate corrective actions, and verifies their implementation. The QCC reviews the laboratory report for correctness, reasonableness, and compliance with project requirements.

3.1.3.1.10 Health and Safety Coordinator (HSC)

The Health and Safety Coordinator (HSC), Mr. Will Max, is responsible for the development of the project HSP and the day-to-day control of health and safety activities. Mr. Max will provide the necessary guidance to the project and laboratory

staffs so they can safely perform their functions in accordance with federal and state regulations.

3.1.3.1.11 Site Safety Officer (SSO)

The Site Safety Officer (SSO), Mr. Charles Schick, is responsible to the HSC for implementation of the HSP. Mr. Schick's responsibilities are:

- Supervise the day-to-day implementation of the site-specific health and safety program
- Train new site personnel on site specific health and safety items
- Interact with project personnel on health and safety matters
- Investigate and report accidents/incidents
- Maintain liaison between field activities, the Project Manager, and the HSC.
- Perform air quality and personal monitoring as required
- Assist the Project Manager in enforcing the requirements of this manual and the site-specific HSP
- Complete all required forms on a timely basis

3.2 Project Communications

Incoming project-related materials in the form of correspondence, sketches, logs, authorizations, or other information shall be routed to the Project Manager after the original is marked with the date received and the project number by a member of the project staff or a secretary assigned this duty. The Project Manager shall then determine which personnel should review the incoming materials and shall route the materials accordingly.

As soon as practical, incoming correspondence originals shall be placed in the project central file. If the correspondence is required by the project personnel for reference, a copy shall be made rather than holding the original. Correspondence which is addressed to the project group but is of importance to the project QA/QC Program shall be routed to the QA officer.

Project-related materials transmitted externally to IT including correspondence, reports, and drawings shall be appropriately reviewed, approved, and if necessary signed prior to transmittal. Outgoing correspondence shall, as a minimum, be signed by the Project Manager or a key level individual assigned this responsibility by the Project Manager. If joint signatures are desirable, the originator of the correspondence, when different than management, may also sign. QA correspondence shall be signed by the Corporate Director of QA or the QA officer. All communications shall be confidential.

Outgoing project correspondence and reports should be read by the Project Manager prior to mailing. The office copy of project correspondence should bear routing information and be routed to QA personnel, if judged appropriate by the Project Manager.

Communications relative to the project which are initiated by third parties (e.g., media, interested individuals, and groups) will be referred directly to EMO without comment.

4.0 QUALITY ASSURANCE PROGRAM

4.1 Quality Assurance Policy

The purpose of the Quality Assurance (QA) Program is to establish policies to facilitate the implementation of regulatory requirements and to provide an internal means for control and review so that work performed is of the highest professional standards.

4.2 Project QA Objectives

This project will be performed in conformance with applicable federal, state, local and contract requirements. Project objectives are as follows:

- Scientific data generated will be of sufficient or greater quality to stand up to scientific and legal scrutiny.
- Data will be gathered or developed in accordance with procedures appropriate for the intended use of the data.
- Data will be of known and acceptable precision, accuracy, representativeness, completeness, and comparability as required by the project.

The QAPP has been prepared in direct response to these goals. The QAPP describes the QA Program to be implemented and the QC procedures to be followed for the WPAFB Field Investigation/Focused Feasibility Study (FI/FFS).

The procedures contained or referred to herein have been taken from the U.S. EPA Quality Assurance Manual EPA/QAMs-005/80 and other appropriate references.

4.3 Analytical QA Objectives For Measurement Of Data

QA objectives have been established for precision, accuracy, completeness, representativeness, and comparability for each major measurement parameter for the WPAFB project. The objectives presented in Tables 4-1 and 4-2 are pertinent to both the IT analytical laboratory and field procedures. All measurements will be representative of the ground water tested. All data will be calculated and reported in units consistent with CLP analytical procedures and other organizations reporting similar data to allow for comparability of data. Definitions for precision, accuracy, completeness, representativeness and comparability are as follows:

- Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the relative standard deviation for replicates. Comparison of duplicate values is best expressed as the relative percent difference (RPD). Various measures of precision exist depending upon the "prescribed similar conditions."

- Accuracy - The degree of agreement of a measurement (or an average of replicate measurements), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.

- Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.

- Representativeness - Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, a process condition, or an environmental concern.

- Comparability - Expresses the confidence with which one data set can be compared to another.

TABLE 4-1

SUMMARY OF METHODS AND QC REQUIREMENTS FOR WATER SAMPLES
WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO
(Page 1 of 3)

Parameter	Method	Analysis Aliquot[1]	Preservation	Detection Limit	Holding Time	Precision	Accuracy
Volatile Organics	Current CLP-SAS SOW Exhibit D (modified)	25 ml	Cool to 4°C HCl to pH<2		Analyze in 14 Days (<24 Hours on Site)	See Table 4-2[2]	See Table 4-2[2]
Acetone				2 ug/L			
Benzene				1 ug/L			
Bromodichlor-omethane				1 ug/L			
Bromoform				1 ug/L			
Bromomethane				2 ug/L			
2-Butanone				2 ug/L			
Carbon disulfide				1 ug/L			
Carbon tetrachlo-ride				1 ug/L			
Chlorobenzene				1 ug/L			
Chloroform				1 ug/L			
Chloroethane				2 ug/L			
Chloromethane				2 ug/L			
cis-1,3-dichloro-propane				1 ug/L			
Dibromochloro-methane				1 ug/L			
1,1-Dichloroethane				1 ug/L			
1,1-Dichloroethene				1 ug/L			
1,2-Dichloroethane				1 ug/L			
1,2-Dichloroethene				1 ug/L			
1,2-Dichloropropane				1 ug/L			

[1] Amount used for single analysis, not amount to collect.

[2] Representative compounds will be used for spikes.

Table 4-1
SUMMARY OF METHODS AND QC REQUIREMENTS
(Page 2 of 3)

Parameter	Method	Analysis Aliquot[1]	Preservation	Detection Limit	Holding Time	Precision	Accuracy
Volatile Organics cont'd							
Ethylbenzene				1 ug/L			
2-Hexanone				2 ug/L			
4-Methyl-2 pentanone				2 ug/L			
Methylene chloride				1 ug/L			
Styrene				1 ug/L			
Tetrachloroethene				1 ug/L			
1,1,2,2-Tetrachloroethane				1 ug/L			
Toluene				1 ug/L			
Trans-1,3-dichloropropane				1 ug/L			
1,1,1-Trichloroethane				1 ug/L			
1,1,2-Trichloroethane				1 ug/L			
Trichloroethene				1 ug/L			
Vinyl acetate				1 ug/L			
Vinyl chloride				1 ug/L			
Xylenes				1 ug/L			
Total Fuel Hydrocarbons	GC/FID[3]		Cool to 4°C				
High Boilers		1 L		50 ug/L[4]	Extract in 14 Days; Analyze in 40 Days	20	40-160
Low Boilers		5 ml		50 ug/L	Analyze in 14 Days	20	40-160

[3] Reference from California Leaking Underground Fuel Tank Field Manual- Guidelines for Site Assessments, Cleanup and Underground Storage Tank Closure, Section 3, Appendix B.

[4] 500 ug/L for lubricating oils.

TABLE 4-1

SUMMARY OF METHODS AND QC REQUIREMENTS
(Page 3 of 3)

Parameter	Analysis Method	Aliquot[1]	Detection Preservation	Holding Limit	Time	Precision	Accuracy
Metals	Current		HNO_3 to pH<2				
Sodium	CLP SOW	100 ml		5,000 ug/L	6 Months	20	75-125
Calcium	200.7 CLP-M	100 ml		5,000 ugL	6 Months	20	75-125
Iron	200.7 CLP-M	100 ml		100 ug/L	6 Months	20	75-125
Magnesium	200.7 CLP-M	100 ml		5,000 ug/L	6 Months	20	75-125
Manganese	200.7 CLP-M	100 ml		15 ug/L	6 Months	20	75-125
Potassium	200.7 CLP-M	100 ml		5,000 ug/L	6 Months	20	75-125
Anions			Cool to 4°C				
Chlorides	9252	50 ml		0.5 mg/L	Analyze in 28 Days	20	75-125
Sulfates	9038	25 ml		2 mg/L	Analyze in 28 Days	20	75-125
Carbonate	310.1	50 ml		1 mg/L	Analyze in 14 Days	20	80-120[5]
Bicarbonate	310.1	50 ml		1 mg/L	Analyze in 14 Days	20	80-120[5]
Others							
Hardness	130.2	50 ml	HNO_3 to pH<2	1 mg/L	6 Months	20	80-120[5]
TDS	160.1	100 ml	Cool to 4°C	2 mg/L	Analyze in 7 Days	20	80-120[5]
Alkalinity	310.1	50 ml	Cool to 4°C	1 mg/L	Analyze in 14 Days	20	80-120[5]
pH	150.1	25 ml	Cool to 4°C	NA	Immediately	20	NA
TOC	9060	25 ml	Cool to 4°C H_2SO_4 to pH<2	3 mg/L	Analyze in 28 Days	20	75-125

[5] For these parameters, blank spikes will be used to assess accuracy.

TABLE 4-2

VOLATILES QUALITY CONTROL LIMITS
WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO

Matrix Spike/Matrix Spike Duplicate

Matrix Spike Compound	%Recovery	Relative Percent Difference
1,1-dichloroethene	61-145	14
trichloroethene	71-120	14
chlorobenzene	75-130	13
toluene	76-125	13
benzene	76-127	11

Surrogates

Surrogate Compound	% Recovery
toluene - d8	88-110
bromofluorobenzene	86-115
1,2 - dichloroethane - d4	76-114

5.0 FIELD SAMPLING PLAN

The Field Sampling Plan (FSP) is included as part of the QAPP in this SAP to eliminate repetition of sampling information and to form a concise document which presents pertinent plans for use by field and management personnel. Draft Standard Operating Procedures (SOPs) prepared by Martin-Marietta for the Base-wide RI/FS as modified to meet the needs of this investigation will be followed for completion of this SAP and are included in Appendix A.

5.1 Field Investigation and Sampling Objectives

The existing data base is incomplete regarding ground-water quality of the Mad River Valley Aquifer at the western boundary of Area C and at the northern boundary of Area B, along Springfield Pike. While organic solvents have been detected in ground water in these areas, the nature and extent of contamination has not been identified to a level sufficient to develop a removal strategy.

Information from the field investigation shall be used to support development of a numerical model to simulate ground-water flow and predict the effectiveness of various control or removal programs. The field investigation at WPAFB has been divided into three separate activities consisting of monitoring well installation, ground-water sampling, and water-level measurement.

5.2 Location and Frequency

5.2.1 Monitoring Wells

Several multiple-well clusters will be installed to determine lateral and vertical water quality conditions. Well clusters will be used to target the most likely contaminant pathways across Area B and C boundaries. To facilitate location of the screened zones for each well in the cluster, a deep pilot hole will be drilled. For the Phase I, Task 4 investigation, the pilot hole will be limited to 150 feet or less to correspond to the primary water supply/production zones of the aquifer.

In order to meet the accelerated schedule and minimum data requirements, eight clusters will be installed during Task 4. Three clusters will be installed in Area B between the West Well Field and the Area B property line. In Area C, five clusters each will be installed along the western property line. New monitoring well locations, shown in Figure 5-1, were selected to evaluate contaminant migration from potential on-Base contaminant sources toward the Rohrer's Island Well Field. Table 5-1 provides general information on the depth of new and existing wells constituting a cluster.

Following installation, each new well will be sampled once for analysis. Analytical results from these wells will be added to the data generated during the resampling of existing wells to provide a three-dimensional representation of ground-water quality.

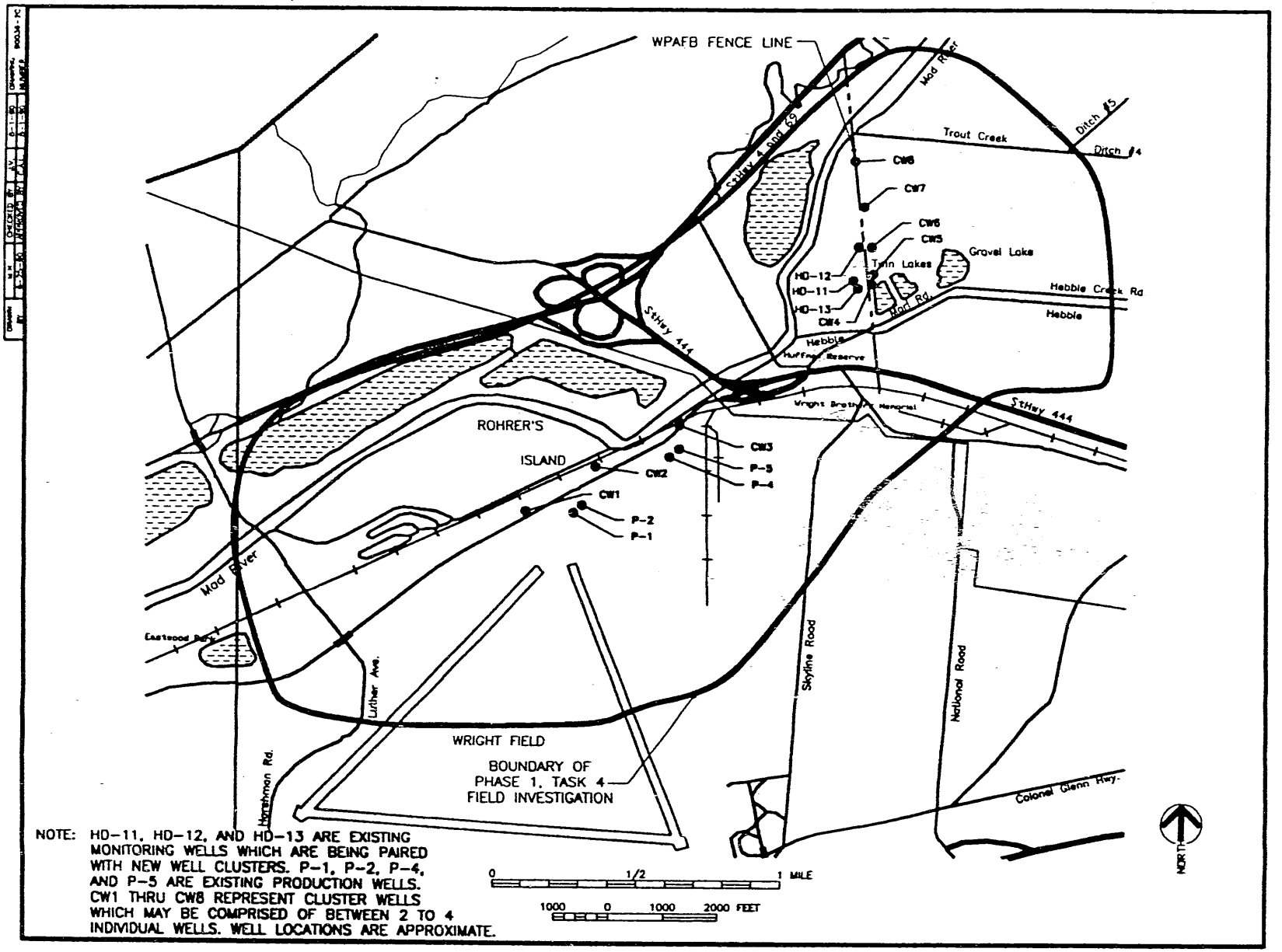


Figure 5-1. Location of New Phase I, Task 4 Monitoring Well Clusters

TABLE 5-1. GENERALIZED WELL-CLUSTER CONFIGURATION

Cluster I.D.	Existing Wells Placed Near	Depth of New Wells[1]	Depth of All Wells in Cluster
CW1	NA	<35	<35
		60-70	60-70
		90-100	90-100
CW2	NA	<35	<35
		60-70	60-70
		90-100	90-100
CW3	NA	<35	<35
		60-70	60-70
		90-100	90-100
CW4	HD-13S		32 (HD-13S)
		50-70	50-70
	HD-13D		107 (HD-13D)
		130-150	130-150
CW5	MW-21		23 (MW-21)
	HD-11		81 (HD-11)
		90-110	90-110
		130-150	130-150
CW6	HD-12S		14.5-24.5 (HD-12S)
	MW-20		21 (MW-20)
	HD-12M		55 (HD-12M)
		70-80	70-80
		100-120	100-120
CW7	08-525-M		16 (08-525-M)
		40-70	40-70
		90-110	90-110
		130-150	130-150
CW8	NA	20-30	20-30
		40-70	40-70
		90-110	90-110
		130-150	130-150

Footnotes

[1] These footages are estimated ranges only and are subject to revision based on knowledge gained during the course of the field investigation.

NA = Not applicable

5.2.2 Ground-Water Sampling

To develop a current picture of conditions at and near the boundaries of Areas B and C, existing monitoring and production wells will be sampled for chemical analysis. All existing wells at the Base boundaries and monitoring and production wells reported to contain detectable organic solvents in the area of investigation will be concurrently sampled and analyzed using consistent analytical and sampling procedures. These wells are located along the western boundary of Area C and in the immediate vicinities of Springfield Pike and the Rohrer's Island well field along Area B. Figure 5-2 shows locations of the existing monitoring wells to be sampled during Phase I Task 4.

Each well will be sampled once during this investigation. To the extent practicable all samples will be collected within one week's time.

5.2.3 Water-Level Measurements

To permit calibration of the ground-water flow model, a complete round of water-level data from existing and new monitoring wells at and near the property boundaries will be collected. This data will act as the baseline against which the numerical or analytical model outputs will be compared.

Figure 5-3 shows the location of monitoring wells from which water-level data will be collected. The investigation will be limited to monitoring wells to avoid problems with pumps and cables in production wells and to eliminate effects of head loss caused in production wells by screen encrustations and well inefficiencies.

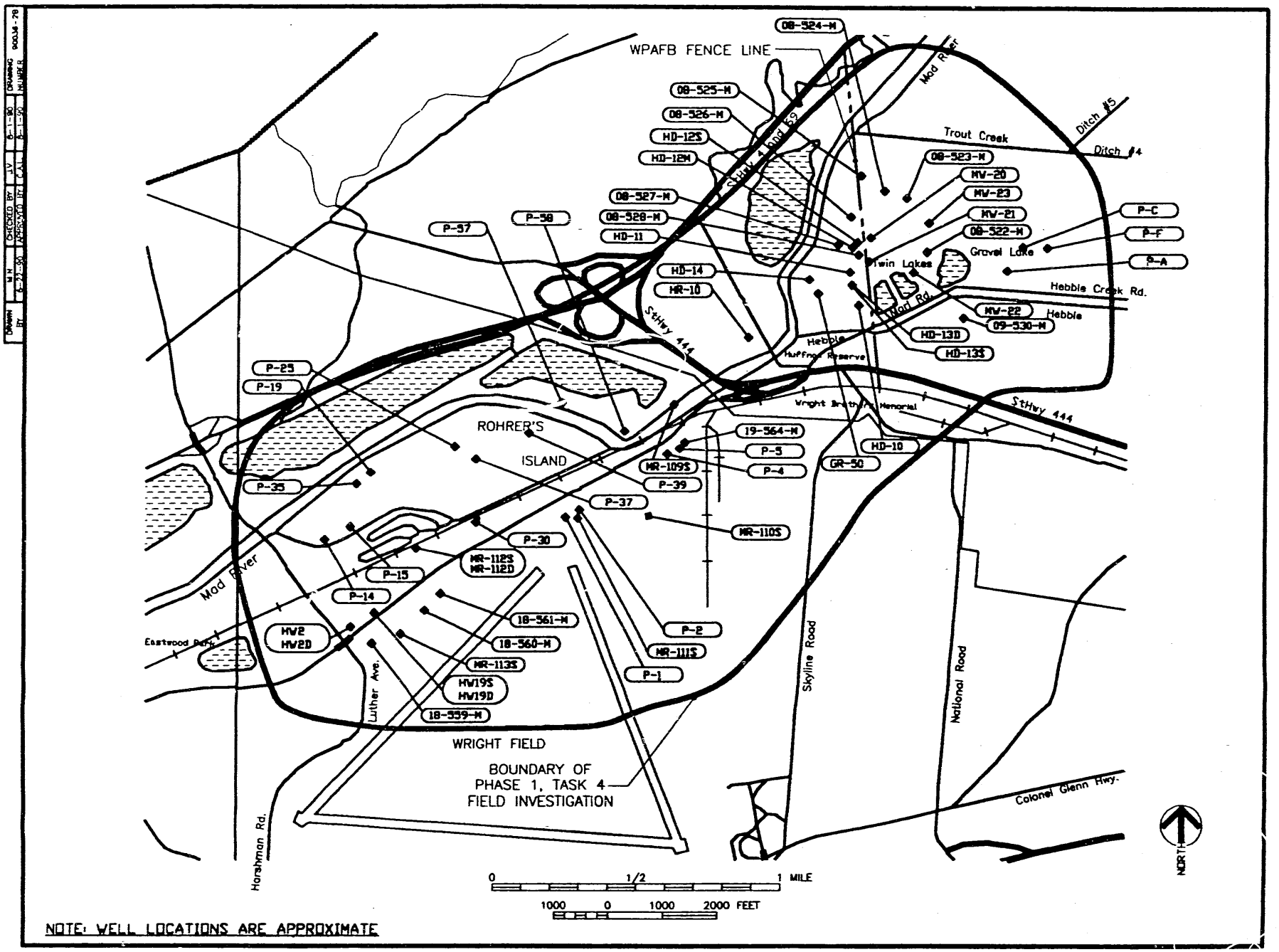


Figure 5-2. Location of Existing Wells to be Sampled for Phase I, Task 4

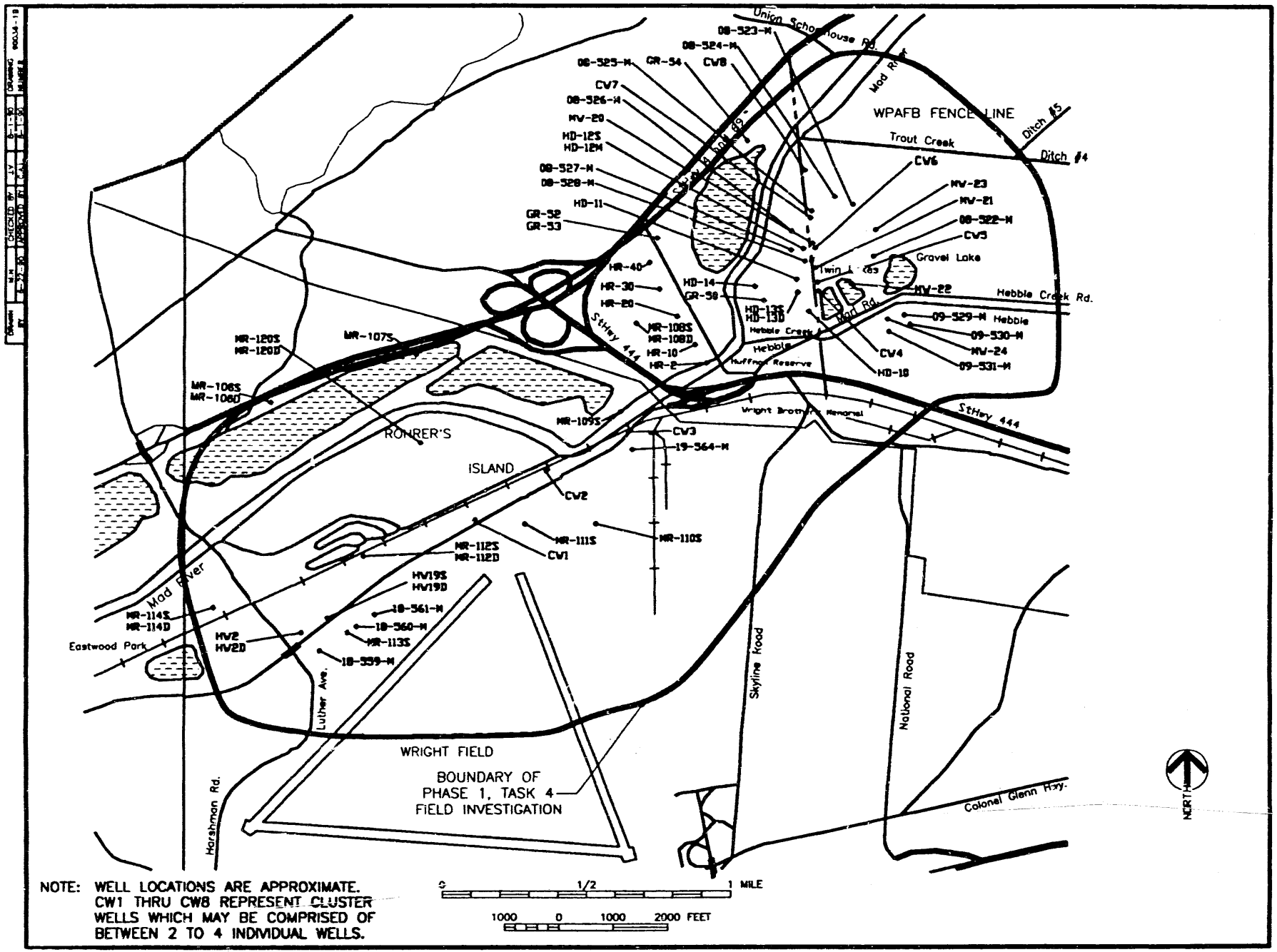


Figure 5-3. Location of Monitoring Wells Where Water Levels will be Measured for Phase I, Task 4

5.3 Sample Identification Nomenclature

A sample nomenclature system will be used to permit easy identification of the sample type and sample location when retrieving data, reviewing analytical results, or performing data manipulation. The nomenclature selected for this project will consist of three unique alpha-numeric codes separated by slashes. The first series of letters and numbers will identify the monitoring well sampled (including the well nest location and relative depth for new wells). The second series in the sample nomenclature will be comprised of numbers only and will indicate the date of sample collection (i.e. 0802 indicates the sample was collected on August 2). The final series of letters in the sample identifier will indicate the purpose for the sample (i.e. whether the sample is an actual sample data point, a matrix spike sample, a matrix spike duplicate, or other QA/QC sample).

In order to prevent any confusion which could be caused by renaming wells previously identified by other contractors, existing monitoring wells designated for resampling will not be renamed. Samples will be identified using the already existing well nomenclature. Modifiers such as the sampling date code and the sample purpose code will be added, however. The ITAS Laboratory will assign a different unique alpha-numeric code for the sample when received by the laboratory to maintain confidentiality of the sample.

5.3.1 Well Identification

The first alpha-numeric series of the sample nomenclature will indicate the location and if nested, the depth of the well sampled. For example, CW1-150 will indicate that the sample was collected from the 150-foot deep well at cluster location 1. The well identification for existing wells slated for sampling will not be changed. For example, MR-110S is an existing well located in Area B of the base.

5.3.2 Sampling Date

The second series of the sample nomenclature will indicate the date the sample was collected. For example, CW1-150/0822 indicates that a ground-water sample was collected from the 150-foot deep well at well cluster location 1 on August 22.

5.3.3 Sample Purpose

The third series of the sample nomenclature will indicate the purpose of the sample. For example, CW1-150/0822/01 indicates the sample is the original or actual sample collected for the study whereas, CW1-150/0822/05 is a matrix spike aliquot collected from the 150-foot deep well at cluster location 1. Each sample type collected during the sampling program will be identified by the following two-digit alpha codes:

01 - Actual Data Sample.
02 - Field Duplicate
03 - Trip Blanks
04 - Equipment Blanks
05 - Matrix Spike
06 - Matrix Spike Duplicate

The following are examples of complete sample identification:

CW2-30/0803/02 Duplicate sample collected at the 30-foot deep well at cluster location 2.

CW5-150/0803/04	The field equipment blank sample collected after sampling well CW5-150.
HD-13D/0804/03	The trip blank sample which was sent along with the sample from well HD-13D

5.4 Monitoring Wells

5.4.1 Drilling Program

Eight well clusters consisting of at least two wells each will be installed in Areas B and C as shown on Figure 5-1 and described in Table 5-1. Three clusters (CW-1, CW-2, and CW-3) will be in Area B between the West Well Field and the Area B property line. In Area C, five clusters (CW-4, CW-5, CW-6, CW-7, and CW-8) will be installed along the western property line. To facilitate location of the screened zones for each well in the nest, a deep pilot hole will be drilled to a maximum depth of approximately 150 feet. Special effort will be made when selecting the screen interval to identify zones of preferential flow.

The pilot hole shall be drilled using cable tool drilling techniques. The SOP for installing boreholes by cable tool is contained in FP5-1 in Appendix A. During cable tool drilling, a minimum 6-inch casing will be advanced as drilling progresses and samples will be collected from cable tool buckets at five-foot intervals. The cuttings will be visually examined by the driller and site geologists for gross changes in lithology each time the bailer is withdrawn from the borehole, and a lithologic sample will be collected (in addition to five-foot interval samples) each time a change in lithology is detected. Cable tool bucket samples will be screened with an FID or PID upon receipt of the sample at the surface. In the event that a well is installed through

a confining layer, the well will be double-cased. Because driving the casing may be difficult through parts of the formation, particularly through the boulder zone, the OEPA's water well standards (OAC 3745-9-06) will be used for assistance in determining which well casing is appropriate for the project (See Table 5-2). No soil samples will be submitted for chemical analysis; however, one soil sample from the screened interval of each well will be submitted for grain-size distribution analysis. Grain-size data will be used to verify visual classification of soils and to provide an indication of anticipated permeability in screened intervals as an aid in designing a remedial system.

Wells constructed to a depth less than 80 feet below grade will be installed in boreholes constructed using hollow stem auger (HSA) drilling techniques. The SOP for installing boreholes with HSA equipment is contained in FP5-1 in Appendix A. The boreholes shall be drilled without sampling, except that one standard split-spoon sample shall be collected in the screened zone for grain-size distribution analysis.

Cable tool and HSA have been selected to minimize the need for drilling fluids. If fluid is needed (above water-table in cable tool holes) potable water from the Base system shall be used. A sample of the drilling fluid will be collected from the water truck for analysis to determine potential contaminants that may be introduced by the drilling fluid. To assure a continuous source of potable water for drilling, the water truck will remain on site throughout the drilling program.

A geologist present during all drilling operations will maintain a detailed boring log for each hole. The log will serve as a record of sample collection, sample location

TABLE 5-2. RECOMMENDED WELL CASING SIZES WITH REGARD TO VARIOUS SOIL FORMATIONS

Casing Recommended for Use Where Hard Driving or Corrosive Waters May Be Encountered (Known as Drive Pipe, Driven Well Pipe, Standard Pipe, Line Pipe, or Reamed and Drifted Pipe)				Casing Suitable for Driving Where Conditions Are Favorable (Known as Water Well Casing)			
Nominal Size in Inches	Outside Diameter in Inches	Wall Thickness in Inches	Weight of Plain End Casing in lbs/ft	Nominal Size in Inches	Outside Diameter in Inches	Wall Thickness in Inches	Weight of Plain End Casing in lbs/ft
1.000	1.315	0.133	1.68	3.500	3.500	0.125	4.51
1.250	1.660	0.140	2.27	4.000	4.00	0.134	5.53
1.500	1.900	0.145	2.72	4.500	4.50	0.142	6.61
2.000	2.375	0.154	3.65	5.500	5.50	0.142	8.13
2.500	2.875	0.203	5.79	6.000	6.00	0.142	8.88
3.000	3.500	0.216	7.58	6.625	6.625	0.156	10.78
3.500	4.000	0.226	9.11	8.625	8.625	0.188	16.94
4.000	4.500	0.237	10.79				
5.000	5.563	0.258	14.62				
6.000	6.625	0.280	18.97				
8.000	8.625	0.277	24.70				
10.000	10.750	0.279	31.20				
12.000	12.750	0.330	43.77				
14.000	14.000	0.375	54.57				
16.000	16.000	0.375	62.58				

Casing of a nominal size not listed in the above table shall have a thickness not less than that required for the next smaller nominal size listed.

and depth, and drilling procedures. The SOP for completing boring logs is contained in FP7-3 in Appendix A.

All drilling and sampling equipment will be decontaminated utilizing SOP's FP3-3 and FP3-1, respectively, which are contained in Appendix A.

5.4.2 Cuttings Disposal and Borehole Abandonment

Cuttings will be screened in the field with a PID or FID, then disposed of in the following manner:

- If reproducible field screening readings indicate volatile organic levels to be less than the instrument's sensitivity or background levels, drill cuttings will be thin spread at the surface in the direct vicinity of the boring.

- If reproducible field screening readings indicate levels of contaminants above background levels, drill cuttings will be collected, covered, and stored in a secure location. Representative soil samples will be collected at the completion of the field program and analyzed for TPH and TCL VOCs. Procedures to be followed in testing and disposing of project-generated wastes will be those identified in the RI/FS Field SOPs and the RI/FS Workplan.

It is anticipated that monitoring wells will be installed in all soil borings; however, in the event that borehole abandonment is necessary per OAC 3745-9-10, the borehole will be abandoned according to the procedures described in SOP FP5-7 in Appendix A. The PID and FID will be operated and calibrated as indicated in SOPs FP2-2 and FP2-3, respectively. These SOPs are contained in Appendix A.

5.4.3 Installation

Monitoring wells will be installed by a qualified drilling subcontractor supervised by an IT geologist. Well installation procedures are found in SOP FP5-2 in Appendix A. Wells will be constructed of 316 2-inch I.D. stainless steel riser with ten foot long, 0.010 inch slot size stainless steel screen. If a confining layer is penetrated (anticipated in Area B), the deep wells will be double-cased with 6-inch mild steel. The annulus between the secondary casing and the borehole shall be grouted in accordance with SOP FP5-2 in Appendix A. At the completion of each boring, well screen and riser will be decontaminated as indicated in SOP FP3-2 contained in Appendix A, assembled and lowered through the auger or casing. Filter pack (washed coarse silica sand) will be placed around the screened interval of each well as augers or casing are correspondingly withdrawn from the hole. The filter pack will extend a maximum of two feet above the top of the screened section. A minimum three-foot thick bentonite seal will be placed above the filter pack. A side-discharging tremie pipe will be used to place the bentonite seal and grout. The bentonite seal will be manually hydrated with potable water if positioned above the saturated zone. The annulus above the bentonite seal will be filled with a cement/bentonite grout as indicated in SOP FP5-2 in Appendix A.

A six-inch I.D. protective steel casing will be installed around each well. Protective casing will be cemented into the ground, fitted with a locking cap with a brass lock, and will display permanent identification. Following well completion, a local

surveyor will provide well top and ground surface elevations so that ground water elevations may be determined.

5.4.4 Well Development

Monitoring wells will be developed in accordance with SOP FP5-4 contained in Appendix A. Development will be conducted by pumping and surging to remove fine-grained materials from the vicinity of the well screen, thereby increasing the hydraulic communication between the well and the aquifer. All wells will be developed until clear water is produced with a minimum amount of suspended sediment as determined during visual inspection of the water and nephelometer readings. Development will continue for a maximum of eight hours; however, if the turbidity requirements are not met after eight hours, samples will be collected and analyzed for silt, clay and total organic carbon according to the procedures described in FP5-4 and the RI/FS Workplan to determine the cause of the turbidity failures. All equipment placed into the well will be decontaminated per SOP FP3-1 in Appendix A prior to use to prevent contamination of wells.

Water removed from wells during development will be collected and stored on site in bulk tanks or portable pools at a location specified by WPAFB. The water will be sampled and analyzed as appropriate for disposal.

5.4.5 Ground-Water Sampling

All newly installed monitoring wells and 52 existing monitoring and production wells will be sampled once during the field program in accordance with SOP FP6-5 contained in Appendix A. New wells will be allowed to stabilize after development for

at least 24 hours. Prior to sampling, each monitoring well will be purged and sampled using the procedures listed below:

- Record well number, sample identification number, date, time, sampling personnel and weather conditions on the sample collection log.

- Remove well cap and screen well head with a PID or FID to determine the presence of volatile organic compounds. Record results on the sample collection log.

- Measure and record the water-level and well depth using an electronic water-level indicator.

- Determine the presence of any floating non-aqueous phase liquid (NAPL) by removing one clear bailer of liquid from the top of the well. Visually inspect the liquid and screen it with a PID or FID to determine whether free product is present. If free product is visibly detected, measure and record the volume of free product present and decant the NAPL into a 40-ml VOA vial for analysis. The EMO Program Director will be notified immediately if this situation occurs during the course of the field investigation.

- Determine the presence of any dense non-aqueous phase liquid (DNAPL) by dropping a bottom-filling bailer to the bottom on the well. Visually inspect the liquid and screen it with a PID or FID to determine whether a "sinker" is present. If free product is visibly detected, measure and record the volume of free product present, and decant the DNAPL into a 40-ml VOA vial for VOC analysis.

- Purge the well as directed in SOPs FB5-5 and FB5-6 contained in Appendix A and record the pH, temperature and conductivity of the ground water. Continue this cycle until three consecutive pH, temperature and conductivity readings are within 10 percent. If the well does not recharge fast enough to purge the necessary well volumes, the well will be pumped dry and sampled as soon as sufficient recharge has entered the well. Purging will be performed in shallow wells utilizing a thoroughly decontaminated stainless steel or Teflon bailer. For deep wells, purging will be performed using an OEPA approved (non-airlift) portable submersible pump.

- Ground-water samples will be collected as indicated in SOP 6-5 in Appendix A. Samples will be collected before any in-line treatment is performed (e.g., activated carbon filter).

- Store samples in a cooler with ice until subsequent preparation and shipment as indicated in SOP FB6-7 contained in Appendix A.

For production wells use the following procedures to collect samples:

- Record well number, sample identification number, date, time, sampling personnel and weather conditions in the sample collection log.

- Locate spigot on well or discharge line at closest proximity to pump.

- If the production well is in operation open spigot and drain at least 5 gallons of water. If the well is not in operation, purge water until field parameters (pH, temperature, and conductivity) stabilize.

- Collect sample by placing sample bottles immediately below spigot. To minimize potential volatilization maintain flow at lowest possible rate. Samples will be collected in order of descending volatility.

- Store samples in a cooler with ice until subsequent preparation and shipment.

All sampling equipment will be decontaminated prior to sample collection according to SOP FP3-1 in Appendix A.

5.4.6 Water-Level Measurements

One round of ground-water level measurements will be taken according to SOP FP7-2, in Appendix A. Water-levels will be taken using a clean electronic water-level indicator on the same day from all wells specified in Section 5.2.3, if possible. If it is not feasible to take the ground-water level measurements on the same day, the water level readings will be collected over a period not to exceed two consecutive days. The probe of the water-level indicator will be decontaminated before each use by the

procedures described in SOP FP3-1 in Appendix A. Water-level measurements will be recorded on field logs for use in determining potentiometric surfaces of the upper and lower flow systems.

5.4.7 QC Sampling

To check the quality of data from field sampling efforts and laboratory handling, blanks and duplicate samples will be collected and submitted to an IT Analytical Services (ITAS) Laboratory for analysis. Blank and duplicate samples will be treated as separate samples for identification, logging and shipping. Analytical results on blank and duplicate samples will be filed with the appropriate field sample data.

Trip blank samples will be used to check for container contamination or contamination from exposure to airborne or ambient contaminants during travel. Trip blanks will consist of VOA vials filled with organic-free water at the laboratory and shipped with empty containers to the project site. One trip blank will accompany VOA vials into the field throughout the sampling effort, will be submitted to the lab with the samples collected for that day, and will remain unopened until the time of analysis. One trip blank will be prepared per shipment (i.e. cooler). Trip blanks will be analyzed for TCL VOCs.

Field/equipment blanks will be prepared to determine whether decontamination procedures are effective in cleaning sampling equipment. The sampling equipment will be thoroughly decontaminated following the procedures outlined in SOP FB3-1 contained in Appendix A. The sampling device will then be rinsed with clean deionized/distilled water. This water will be collected in appropriate sample

containers. One field blank will be prepared each day for each sampling team at the site and analyzed for TCL VOCs.

Duplicate or split samples will be used to evaluate the precision of both the collection and analytical procedures. Duplicate sampling is defined as two samples collected for the same analytical parameter from the same location. One duplicate sample will be collected for every 10 samples collected at the site (10% duplicate sampling rate) and analyzed for the same parameters as the original sample.

The CLP SOW requires matrix spike (MS) and matrix spike duplicate (MSD) analysis at a rate of one MS/MSD pair per 20 samples (5% MS/MSD sampling rate). When an MS/MSD pair is to be analyzed, additional sample must be collected. The sample is then split at the laboratory and spiked to determine the accuracy and precision of the laboratory analytical procedure.

5.4.8 Decontamination Procedures

Decontamination of sampling equipment in direct contact with the sample and equipment that does not directly contact the sample (e.g., HSAs, temporary casing, and drill rod) is addressed in the following subsections.

Two decontamination areas will be setup for the Task 4 field investigation. One will be in Area A and one will be in Area B. Decontamination fluids will be collected and properly handled in accordance with Federal, State, and local regulations.

5.4.8.1 Equipment in Direct Contact with Samples

Decontaminating sampling devices in direct contact with the sample will be conducted according to procedures described in SOP FP3-1 (Appendix A). All

decontamination fluids will be collected and transported for final disposal through WPAFB.

5.4.8.2 Equipment Not in Direct Contact with Samples

Sampling equipment not in direct contact with samples such as HSAs, temporary casing, drill rods and drilling rigs will be decontaminated between samples or boreholes by procedures described in SOP FP3-3 (Appendix A).

5.5 Sampling Handling and Analysis

All samples collected for chemical analysis during this project are assumed to be classified as low-level hazard rated samples or environmental samples. Procedures for low-level samples are described below and in SOP FB6-7 in Appendix A.

5.5.1 Sample Preparation

As each sample is collected in the field, it will be placed in a labeled sample bottle with appropriate preservatives and stored in an iced cooler as indicated in SOP FB6-7 contained in Appendix A. Sample preparation will include hand tightening all bottle lids, properly labeling and tagging each sample and storing them on ice at 4°C. Chain-of-custody documents will be prepared for all samples which will be shipped to a laboratory.

5.5.2 Sample Bottle Preparation and Sample Preservation

Sample containers and appropriate preservatives are presented in Table 5-3 for ground-water samples for specific chemical parameters. Samples requiring preservation shall be preserved in the field with the appropriate reagents supplied by

TABLE 5-3
WATER SAMPLING SPECIFICATIONS

Parameter	Container[1]	Sample Preservation	Holding Time
Volatile Organics	Two 40-ML Glass Vials, No Headspace	HCl to pH<2 Using 3 drops HCL Immediately After Sampling Ice to 4°C	Analyze in 14 Days
Total Petroleum Hydrocarbons	One 1-liter Glass Bottle	Ice to 4°C	28 Days
Metals and Hardness (Na,Ca,Fe,Mg, Mn, K)	One 1-liter Plastic	HNO_3 to pH< 2 Ice to 4°C	6 months
Anions, TDS, Alkalinity, and pH	One 1-liter Plastic	Ice to 4°C	28 Days for Chlorides and Sulfates; 14 Days for Carbonate and Bicarbonate; 7 Days for TDS; 14 Days for alkalinity; pH test required immediately upon collection
TOC	500-mL Glass	Ice to 4°C H_2SO_4 to pH<2	28 Days

1 All Containers will have teflon lined lids (Septa for VOAs) and be EPA approved.

the analytical laboratory. Sample bottles, such as I-Chem Protocol A, are purchased precleaned in accordance with U.S. EPA specifications.

5.5.3 Storage and Shipping

Samples which will be delivered to the laboratory for analysis will be prepared for shipment using the following procedures:

- Tighten sample bottle lids hand tight. Place custody tape over lid and sample label.
- Place about three inches of packing material in the bottom of a waterproof cooler.
- Seal bottles in clear plastic bags and place in a cooler in such a way that they do not touch.
- Place blue ice in plastic bags and arrange in cooler on and around bottles.
- Fill cooler with packing material.
- Place paperwork in plastic bags and tape to the inside cooler lid.
- Tape drain shut.
- Close cooler and secure lid by taping cooler completely around the outside with strapping tape at two locations.
- Place lab address on top of cooler.
- Place "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of the cooler.
- Affix custody seals on front right and back left corners of the cooler. Cover seals with wide clear tape as appropriate.
- The proximity of WPAFB to the ITAS Cincinnati laboratory will permit hand delivery of the samples by IT personnel.

All samples will be preserved on the day they are collected and will be shipped on the same day as sample collection.

After the samples have been packaged for shipping, custody will be transferred to a team member who will deliver them directly to ITAS Cincinnati.

5.5.4 Analysis

Table 5-4 is a list of analytical parameters for this investigation. All groundwater samples will be analyzed for TCL volatile organic compounds (VOCs) using Contract Laboratory Program Special Analytical Services (SAS) Scope of Work Methods in the most recent CLP SAS SOW and for total petroleum hydrocarbons (TPH) using the GC/FID method described in the California Leaking Underground Fuel Tank Field Manual - Guidelines for Site Assessments, Cleanup, and Underground Storage Tank Closure.

Samples shall be collected from three new monitoring wells in Area B and three new monitoring wells in Area C for analyses necessary to evaluate above-ground treatment facilities. Each sample shall be analyzed for metals (e.g. calcium, sodium, iron, potassium, magnesium, and manganese), anions (e.g. chloride, sulfate, and carbonate/bicarbonate), hardness, alkalinity, total dissolved solids, pH, and TOC.

ITAS SOP's for equipment calibration, sample preparation, analyses, and decontamination are included as Appendix B.

TABLE 5-4 LIST OF ANALYTICAL PARAMETERS

TCL Volatile Organic Compounds

Acetone
Benzene
Bromodichloromethane
Bromoform
Bromomethane
2-Butanone
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chloroform
Chloroethane
Chloromethane
cis-1,3-dichloropropane
Dibromochloromethane
1,1-Dichloroethane
1,1-Dichloroethene
1,2-Dichloroethane
1,2-Dichloroethene
1,2-Dichloropropane
Ethylbenzene
2-Hexanone
4-Methyl-2 pentanone
Methylene chloride
Styrene
Tetrachloroethene
1,1,2,2-Tetrachlorethane
Toluene
Trans-1,3-dichloropropane
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Vinyl acetate
Vinyl chloride
Xylenes

Metals

Calcium
Iron
Manganese
Magnesium
Potassium
Sodium

Anions

Chlorides
Sulfates
Carbonate
Bicarbonate

Miscellaneous Parameters

Alkalinity
Hardness
TDS
TOC
TPH

6.0 CHAIN-OF-CUSTODY

6.1 Field Sampling

The following forms will be used to document chain-of-custody procedures for sample tracking and field activities:

- Sample identification and labeling.
- Sample chain-of-custody.
- Sample collection logs.
- Laboratory request for analysis forms.

6.1.1 Sample Identification and Labeling

All samples will be marked for identification from the time of collection and packaging through shipping. Marking will be done on the sample container (jar, bottle, etc.). Sample identification will include, as appropriate the information in the example standard sample label as illustrated in Figure 6-1.

6.1.2 Chain-of-Custody Records

Documentation of the sample chain-of-custody is provided by the use of chain-of-custody forms which record the sampling location, the type and amount of sample collected, the date and time of sample collection, the signature(s) of the person(s) responsible for sample collection, the date and time of all custody transfers, signature(s) of the person(s) relinquishing and accepting sample custody, and other pertinent information.

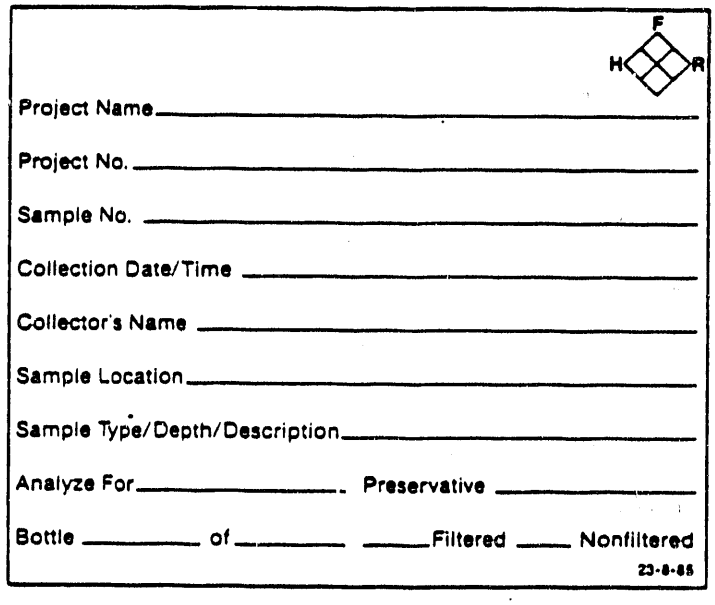


Figure 6-1 Standard Sample Label

Chain-of-custody procedures document sample possession from the time of collection to disposal. A sample is considered in custody if it is:

- In one's actual possession.
- In view, after being in physical possession.
- Locked so that no one can tamper with it, after having been in physical custody.
- In a secured area, restricted to authorized personnel.

A chain-of-custody record will be initiated in the field and will accompany each group of samples during shipment to the laboratory.

Each time responsibility for custody of the sample changes, the new custodian will sign the record and indicate the dates of transfer. Copies of the signed record will be made by the immediately previous custodian and sent to the project manager and the sample custodian at the IT laboratory to advise them of any change in the status of the sample. An example of an IT chain-of-custody record is included in Figure 6-2.

6.1.3 Sample Collection Logs

Sample collection logs are filled out for each sample to record information pertaining to the location, condition, and collection of field samples. The following is required on the field collection report form:

- Project name and number.
- Date and time sample collected.
- Field engineer/scientist responsible for sample collection.
- Sample identification number and type, (i.e., water).
- Field testing results in pH, temperature, specific conductance, etc.

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. 57252

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions: _____

Possible Sample Hazards: _____

SIGNATURES: (Name, Company, Date and Time)

1. Relinquished By: _____

   Received By: _____

2. Relinquished By: _____

   Received By: _____

3. Relinquished By: _____

   Received by: _____

4. Relinquished By: _____

   Received By: _____

WHITE - To accompany samples
YELLOW - Field copy

Figure 6-2 Standard Chain-of-Custody Form

- Location sketch of sample collection area.
- Weather conditions.
- Water-level depth collected if monitoring well.
- General field observations.

An example of an T sample collection log is included in Figure 6-3.

6.1.4 Laboratory Request for Analysis Form

Laboratory request for analysis forms are filled out to indicate the testing program required for the collected samples. The following pertinent information is recorded on the laboratory request for analysis forms:

- Project name and number.
- Date samples shipped, the carrier, and the waybill number.
- Required report date and turnaround times for analysis.
- Contact with telephone number for receipt of the analytical report and billing invoices.
- Sample identification numbers.
- Sample volume collected and appropriate preservatives.
- Requested analyses and laboratory methods.

An example of a laboratory request for analysis form is included in Figure 6-4.

DATE						
TIME						
PAGE ____ OF ____						
PAGE						
PROJECT NO.						

SAMPLE COLLECTION LOG

PROJECT NAME _____

SAMPLE NO. _____

SAMPLE LOCATION _____

SAMPLE TYPE _____

COMPOSITE _____ YES _____ NO

COMPOSITE TYPE _____

DEPTH OF SAMPLE _____

WEATHER _____

CONTAINERS USED	AMOUNT COLLECTED

COMMENTS:

PREPARED BY: _____

Figure 6-3 Standard Sample Collection Log

REQUEST FOR ANALYSIS

R/A Control No. 185477

C/C Control No. _____

PROJECT NAME _____

PROJECT NUMBER _____

PROFIT CENTER NUMBER _____

PROJECT MANAGER _____

BILL TO _____

_____

_____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____

LAB DESTINATION _____

LABORATORY CONTACT _____

SEND LAB REPORT TO _____

_____

_____

DATE REPORT REQUIRED _____

PROJECT CONTACT _____

PROJECT CONTACT PHONE NO. _____

Sample No	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Laboratory Project Manager.)

Normal _____ **Rush** _____ (Subject to rush surcharge.)

QC LEVEL: (Levels II and III subject to surcharge; project-specific requirements must be submitted to lab before beginning work.)

I _____ **II** _____ **III** _____ **Project Specific** _____

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances.)

Non-hazard _____ **Flammable** _____ **Skin Irritant** _____ **Highly Toxic** _____ **Other** _____ **(Please Specify)**

SAMPLE DISPOSAL: (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping, archive and disposal.)

Return to Client _____ **Disposal by Lab** _____ **Archive** _____ **(Indicate number of months.)**

FOR LAB USE ONLY

Received by _____ **Date/Time** _____

WHITE - Original, to accompany samples

YELLOW - Field copy

Figure 6-4 Standard Request-for-Analysis Form

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6.2 Analytical Laboratory

6.2.1 Laboratory Sample Receipt

The Operations Manager shall note that the shipment is expected and notify the operations manager or group leaders of the incoming samples. Upon sample receipt, the Sample Control personnel shall:

- Examine all samples and determine if proper temperature has been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be considered damaged. It will be noted on the chain-of-custody record that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible that samples that were damaged and that they must be resampled, or the testing program changed, and an estimate of the cause of damage.

- Compare samples received against those listed on the chain-of-custody record.

- Verify that sample holding times have not been exceeded.

- Sign and date the chain-of-custody form and attach the waybill to the chain-of-custody.

- Enter the samples in the laboratory sample log-in book which contains the following information:

  - Project identification
  - Sample numbers
  - Type of samples
  - Date received in laboratory
  - Date put into storage after analysis is completed
  - Date of disposal.

The last two items will be added to the log when the action is taken:

- Notify the operations manager or group leaders of sample arrival.

- Place the completed chain-of-custody records in the project file.

If samples arrive without chain-of-custody or incorrect chain-of-custody records, the following shall be done by the Sample Control personnel:

- If the chain-of-custody form is incorrect, a memorandum to the project manager and field personnel is prepared stating the inaccuracy and correction. The memorandum must be signed and dated by the person originating the chain-of-custody and the Sample Control Group Leader. The memorandum will serve as an amendment to the chain-of-custody. If the information on the chain-of-custody form cannot be corrected by the Sample Control Group Leader or the field personnel, the sample affected shall be removed from the sampling program.

- If the chain-of-custody form is not shipped with the samples, the field personnel shall be contacted and a memorandum prepared which lists the person involved in collecting, shipping, and receiving the samples and the times, dates, and events. Each person involved must sign and date this memorandum. The completed memorandum will be maintained in lieu of the chain-of-custody.

6.2.2 Laboratory Storage of Samples

The primary considerations for sample storage are:

- Maintaining the prescribed temperature, if required, which is typically 4°C.

- Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

The requirements for temperatures and holding times prescribed in Section 5.0 shall be used. Placing of samples in the proper storage environment is the responsibility of the Sample Control personnel, who should notify the operations manager or group leaders if there are any samples which must be analyzed immediately because of holding time requirements.

6.2.3 Initiation of Testing Program

As stated in Section 6.1.4, a request for analysis form shall be submitted with samples to the laboratory. If the analytical program is not defined with the sample shipment, the Sample Control personnel shall immediately notify the manager responsible for the work for definition of the analysis program. The analytical program shall be entered in the project log-in form.

The operations manager and group leaders are responsible for prioritizing samples on the basis of holding time and required reporting time.

6.2.3.1 Project Control Boards

Following the completion of the log-in process, the defined analytical testing program will be outlined on the appropriate sample control board in the laboratory. Project name and number are used to identify the specified number of samples to be completed within a required time period. Holding times and due dates are recorded to assist with prioritizing analyses.

6.2.3.2 Data Summary Forms

Following the posting of the analytical testing program on the sample control boards, the testing program will, as necessary, be summarized on project data summary sheets in file folders in the laboratory. These folders will serve as a receptacle for completed laboratory data sheets as analyses are finished by individual analysts.

6.2.4 Sample Disposal

The chain-of-custody for the sample is completed as part of sample disposal. There are several possibilities for sample disposition:

- The sample may be completely consumed during analysis or disposed of by the laboratory.
- Samples may be returned to the client or location of sampling for disposal.
- The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. If reanalysis is not anticipated, environmental conditions for storage will not be observed.

The operations manager shall determine disposition of samples if not specified on the request for analysis form (Figure 6-4). In general, ITAS will not maintain samples and extracts longer than 30 days beyond completion of analysis, unless otherwise specified.

7.0 CALIBRATION PROCEDURES AND FREQUENCY

Measuring and test equipment used in the field and laboratory shall be controlled by a formal calibration program. The responsibility for the calibration of laboratory and associated reference equipment rests with the laboratory managers. Calibration of other equipment is the responsibility of the office or group maintaining that equipment.

Analytical laboratory equipment will be maintained and calibrated in accordance with referenced U.S. EPA CLP analytical procedures as summarized in Table 7-1.

Calibration procedures specific to the analytical methods referenced in Table 4-1 are discussed with the appropriate analytical procedures in Section 8.0. Field equipment will be maintained and calibrated in accordance with the manufacturer's recommended procedures. Field instruments will include a pH meter, thermometer, m-scope, specific conductivity meter, Organic Vapor Analyzer (OVA) or Organic Vapor Photoionization Detector (PID).

The pH meter will be calibrated with standard buffer solutions prior to field use. In the field, the meter will be calibrated before purging of each well. Fresh National Bureau of Standards (NBS) traceable buffer solutions will be used for calibration during each field use. Operation and calibration procedures for the pH meter are outlined in SOP FB7-4 in Appendix A.

The thermometer is calibrated by the manufacturer.

TABLE 7-1
ENVIRONMENTAL LABORATORY
CALIBRATION AND MAINTENANCE PROCEDURES

Instrument/Equipment	Manufacturer and Model	Calibration Method/Procedure	Reference	Calibration Frequency	Maintenance Schedule
Inorganic Analysis					
Inductively Coupled Plasma Emission Spectrometer (ICP)	Perkin-Elmer Plasma II	200.7 CLP-M	(1)	Each Use	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual, and annual activities. Also service contract support.
UV/VIS Spectrophotometer	Baush & Lomb Spectronic 70	9038	(4)	Daily Check	Instrument response and waveiength checked monthly.
Analytical and Top-Loading Balance	Mettler Models PC-4400, HL32, H35	Section 6.2.1[2]	(2)	Quarterly	Routine preventive maintenance program, annual visit by service personnel for calibration.
Organic Analysis					
Gas Chromatograph/ Mass Spectrometer (GC/MS)	Extrell Model ELQ 400 or Finnigan 5100	Exhibit D[3]	(1)	Daily Check	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual and annual activities.
Gas Chromatograph (GC)	Varion 3400	Table 4-2[4]	(3)	Daily Check	Complete routine preventive maintenance program exists with daily, weekly, monthly, quarterly, semiannual and annual activities.
pH Meter	Orion 701A	150.1	(5)	Each Use	Electrode cleaned monthly or as needed.
TOC	Xertex/Dohrman DC-10	9060	(4)	Each Use	Routine preventive maintenance program.

Table 7-1
(continued)

Footnotes:

[1]Generally, the calibration procedure consists of constructing a four-point standard curve consisting of a blank and three calibration standards within the instrument's working range. After the ICP or AA systems have been initially calibrated, the accuracy of the initial calibration is verified and documented for each metal by analysis of an initial calibration verification solution prepared from a source different from the calibration standards. Continuing calibration verification is performed every ten samples or two hours whichever is more frequent as well as at the beginning and end of a sample run. The initial and continuing calibration verification solution (standard) must read within 90-110 percent of its true value. A calibration blank is analyzed each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10 percent during the run. The calibration blank must be below the analytical method detection limit.

[2]Balances are checked with a single weight before each use. They are calibrated quarterly over a range to 95% capacity.

[3]Initial calibration of the GC/MS consists of a five-point standard curve at Contract Laboratory Program (CLP) prescribed concentration levels. The minimum average response factor and the percent relative standard deviation of the response factors must be within acceptance criteria. Continuing calibration must be performed every 12 hours using a single standard and acceptance criteria must be met or the five-point standard calibration must be repeated.

[4]The GC must be calibrated with a minimum of three standards and a blank verified on each working day. If acceptance criteria are not met, a new curve is prepared.

References:

(1) USEPA Contract Laboratory Statement of Work, current revision at time analysis performed.

(2) ITAS Quality Assurance Manual

(3) Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup and Underground Storage Tank Closure.

(4) SW-846, Third Edition

(5) EPA Chemical Analysis of Water and Wastes

The specific conductivity meter will have its conductivity cells cleaned and checked against known conductivity standards before each use in the field. In the field, the instrument will be checked daily with NIST traceable standards. Operation and calibration procedures for the specific conductivity meter are outlined in SOP FB7-5 in Appendix A.

The OVA and PID will be calibrated daily with a gas of known concentration. Results of the field calibration shall be recorded on the sample collection log prepared for each sample. Operation and calibration procedures for these instruments are outlined in SOPs FB2-3 and FB2-2, respectively. These SOPs are contained in Appendix A.

8.0 LABORATORY ANALYTICAL PROCEDURES

The laboratory analytical procedures required for the SAP and are described in the following sections.

8.1 Laboratory Program Flow Chart

The generation of project analytical data and results will follow the standard IT laboratory analytical program management scheme (IT Analytical Services Quality Assurance Manual). The laboratory analysis flow chart (Figure 8-1) outlines the management scheme which consists of five major areas:

- Project initiation
- Handling of collected samples
- Laboratory testing program initiation
- Data verification
- Report preparation

These areas are described in the following sections.

8.1.1 Project Initiation

Prior to initiation of laboratory testing, a planning session with the appropriate laboratory and project staffs will be conducted to discuss the specific aspects of the following project tasks which must be completed at this time:

- Define project requirements, including equipment, parameters, sampling procedures (Section 5.0), QC samples, and analytical methods (Section 8.3) selection.
- Request sample bottles from laboratory custodian.

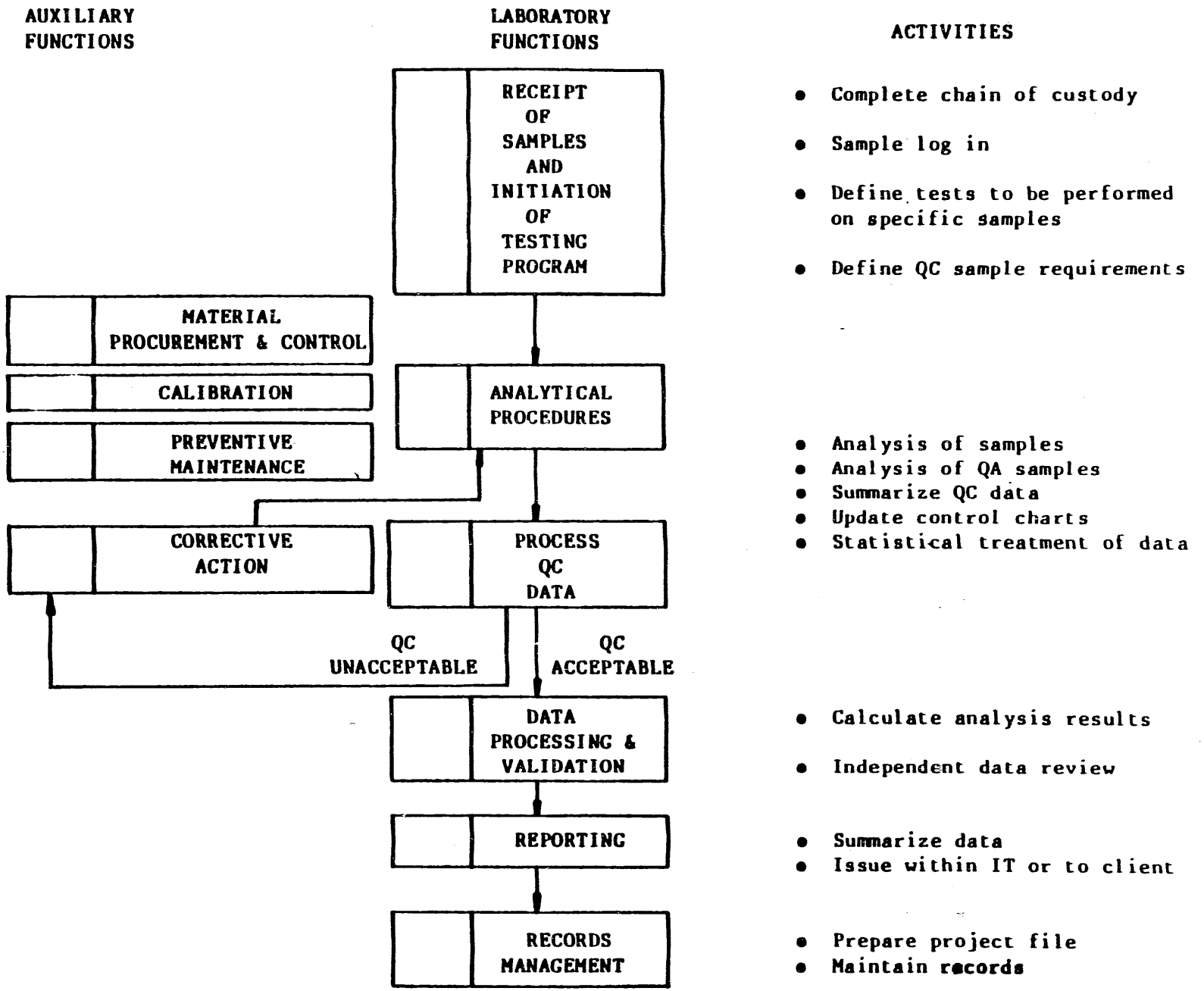

FIGURE 8-1 FLOW CHART OF LABORATORY ACTIVITIES

- Prepare bottles with appropriate labels and preservatives (Sections 5.6 and 6.1.1).

- Provide blank chain-of-custody and request for analysis forms with sample bottles, which will be shipped to the site.

8.1.2 Sample Processing Procedures

The following procedures are implemented by field and laboratory personnel for handling of collected samples:

- Summarize field data collection on field sheets and initiate chain-of-custody forms.

- Transport collected samples to the laboratory under suitable environmental conditions.

- Follow U.S. Department of Transportation (DOT) regulations for sample transport.

- At the laboratory, review received samples and process chain-of-custody forms. Assign samples a unique number upon receipt in the laboratory.

- Place samples in proper storage.

- Log-in samples in laboratory log.

- Log-out samples for analysis.

8.2 Laboratory Testing Program Initiation

8.2.1 Receipt of Samples

Upon receipt, the laboratory sample custodian shall:

- Examine all samples and determine if proper temperature has been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall be also considered damaged. It will be noted on the chain-of-custody record that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible via

an on-site telephone that samples were damaged and that they must be resampled or the testing program changed.

- Compare samples received against those listed on the chain-of-custody.
- Verify that sample holding times have not been exceeded.
- Sign and date the chain-of-custody form and attach any waybill to the chain-of-custody.
- Place the samples in appropriate laboratory storage.
- Enter the samples in the laboratory sample log-in book which contains the following information: project identification number, sample numbers, type of samples, date received in laboratory, date put into storage after analysis is completed, date of disposal.

  The last two items will be added to the log when the action is taken.
- Notify the laboratory director or operations manager of sample arrival.
- Place the chain-of-custody records in the laboratory project file.

8.2.2 Project Control Boards

Following the completion of the log-in process, copies of the work orders are distributed to the appropriate group leaders. The defined analytical testing program will be outlined on the appropriate sample control board in each laboratory area. Project name and work order number are used to identify the specified number of samples to be completed within a required time period. Project status can be monitored from analysis to data processing to review.

8.2.3 Data Summary Forms

For each analysis, data are summarized and reviewed by the appropriate team/group leader. The approved data are then forwarded to the reporting group for inclusion in the final report.

8.3 Laboratory Testing Procedures

Samples from the WPAFB site will be analyzed using the analytical methods listed in Table 4-1. The actual references are maintained in the laboratory for use by laboratory personnel.

The current project-specific laboratory program for WPAFB is presented in Section 5 of this SAP. Subsequent project activities may require revision of the laboratory program. Should this occur, the Work Plan will be formally revised and will become the controlling document for the testing program.

8.4 Analytical Methods

The analytical testing program for the samples collected from the site can be broadly divided into five analytical categories:

- Sample preparation
- Organic analysis
- Metals analysis
- Anions analysis
- Hardness, TDS, Alkalinity, pH, and TOC

8.4.1 Sample Preparation

Sample preparation procedures are described with the appropriate analytical procedures under Sections 8.4.2 and 8.4.3.

8.4.2 Organic Analysis

The organic analyses to be performed include:

- Total Petroleum Hydrocarbons
- TCL Volatile Organics

The analytical program for the total petroleum hydrocarbons will follow the methodologies outlined in the California Leaking Underground Fuel Tank Manual.

The analytical program for the volatiles will follow the methodologies outlined under Exhibit D in the CLP SAS SOW revision which is current at the time analyses are performed. This will include the following conditions:

1. Scans will be conducted for the TCL compounds identified in Table 4-1 of this QAPP.

2. Unknown peaks with heights greater than 10 percent of the nearest internal standard will be tentatively identified by a library search of the U.S. EPA/National Institute of Health (NIH) mass spectral library.

8.4.2.1 Total Petroleum Hydrocarbons

8.4.2.1.1 Method Summary

Total petroleum hydrocarbons may be determined as volatile (gasoline) or semivolatile (diesel) organic compounds. The volatiles are determined by purge and trap GC/FID. The semivolatile hydrocarbons are first extracted with solvent; the extract is then injected into the GC/FID.

8.4.2.1.2 Interferences

Method blanks are used to demonstrate that solvents, reagents and glassware are free from interferences.

8.4.2.1.3 Instrumentation and Operating Conditions

A Varian 3400 GC with FID will be used for the analysis of total petroleum hydrocarbons. The separating conditions will be those recommended in the California Leaking Underground Fuel Tank Field Manual.

8.4.2.1.4 Materials and Reagents

All volumetric glassware will be Class A. Solvents will be high purity.

8.4.2.1.5 Standards

Commercial diesel standards are diluted in carbon disulfide for high boiler analysis. Commercial gasoline standards are diluted in dodecane for low boiler analysis. Calibration standards are prepared at three concentration levels. One should be near but above the detection limit; the others should correspond to the expected concentrations in the samples or define the working range of the detector.

8.4.2.1.6 Analysis

The following briefly describes the procedure for sample analysis:

- Set the instrument with the proper operating conditions.
- Calibrate the instrument with three standards and blank.
- Use an independently prepared reference standard to verify the calibration.
- Check instrument stability by analyzing a calibration standard after every 10 samples.

8.4.2.1.7 Quality Control

Analyze a matrix spike/matrix spike duplicate pair for every 20 samples for both the low boiling and high boiling methods.

8.4.2.2 Volatile Organics

8.4.2.2.1 Method Summary

An inert gas is bubbled through a 5 milliliter water sample which is contained in a specially designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer. The TCL volatile compounds are identified in Table 4-1.

8.4.2.2.2 Interferences

Impurities in the purge gas, organic outgassing the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems.

Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage.

Contamination by carry-over can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carry-over, the purging device and sample and purging of the entire systems may be required after unusually high amounts of volatiles are analyzed.

8.4.2.2.3 Instrumentation and Operating Conditions

The operating conditions used to perform TCL volatile compound analyses will follow CLP SAS and the manufacturer's recommendations.

8.4.2.2.4 Materials and Reagents

All volumetric glassware will be Class A. Chemical reagents will be of high purity.

8.4.2.2.5 Standards

Analytical standards will be commercially purchased and demonstrated to be traceable to National Institute of Standards and Technology (NIST) standards.

- Working Standards - The working standard is prepared by adding the appropriate amount of standard into 5 milliliters of deionized water. Initial calibration is required at 20, 50, 100, 150, and 200 ug/l per each compound. Continuing calibration requires the analysis of a 50 ug/l composite standard.

- Internal and Surrogate Stock Standards - These are commercially purchased. Three surrogates and three internal standards are used. Surrogate compounds are 1,2-dichloroethane-d_4, p-bromofluorobenzene, and toluene-d_8. Internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5.

- Volatiles Matrix Standard Spiking Solution - Prepare a spiking solution in methanol that contains the following compounds

  - 1,1-Dichloroethane
  - Trichloroethene
  - Chlorobenzene
  - Toluene
  - Benzene.

8.4.2.2.6 Calibration

The following procedures summarize the GC/MS calibration process:

- Tune GC/MS system.
- Calibrate using the five calibration standards.
- Calculate relative response factors (RRF) and evaluate calibration and system performance check compound responses.

8.4.2.2.7 Quality Control

Add surrogates to all blanks and samples. Analyze a matrix spike/matrix spike duplicate pair as described in the CLP SOW. This must be done at a frequency of one pair for every 20 samples.

8.4.2.2.8 Calculations

Calculate the compound concentrations in the sample as determined using the following equations from the CLP methods:

$$\text{Concentration ug/l} = \frac{(A_x)(I_s)}{(A_{is})(RRF)(V_o)}$$

where

A_x = area of the characteristic ion for the compound to be measured,

A_{is} = area of the characteristic ion for the internal standard,

I_s = amount of internal standard injected in nanograms,

V_o = volume of water extracted in milliliters,

RRF = the relative response factor calculated for the compound of interest.

8.4.3 Inorganic Analysis

The inorganic analytical procedures which will be used to perform the laboratory program are specified below for:

- Metals
- Chloride
- pH
- Sulfate
- Alkalinity
- TOC
- Hardness
- TDS

8.4.3.1 Metals Analyses

Metals analyses will be performed by inductively coupled argon plasma emission spectrometry (ICP). A summary of the referenced analytical procedures (Table 4-1) is presented below.

8.4.3.1.1 Inductively Coupled Argon Plasma Spectrometry - Method 200.7 CLP-M

8.4.3.1.1.1 Method Summary

The method describes a technique for the simultaneous or sequential multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-like emission spectra are produced by a radio-frequency ICP. The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A

background correction technique is required to compensate for variable background contribution to the determination of trace elements.

8.4.3.1.1.2 Instrumentation and Operating Conditions

A Perkin-Elmer Plasma II ICP spectrometer will be used for ICP analyses. Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain QC data confirming instrument performance and analytical results.

8.4.3.1.1.3 Materials and Reagents

Acids used in the preparation of standards and for sample processing must be ultrahigh purity grade or equivalent. Redistilled acids are acceptable. Volumetric glassware will be Class A.

8.4.3.1.1.4 Standards

Commercially available certified standards will be used.

8.4.3.1.1.5 Analysis

The following summary briefly describes the procedure for analysis of samples:

- Set up instrument with proper operating parameters established in Section 8.4.3.1.1.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.

- Initiate the appropriate operating configuration via the computer.

- Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using mixed calibration standard solutions such as those described in Section 8.4.1.1.1.4. Flush the system with the calibration blank between each standard. Use the average intensity of duplicate exposures for both standardization and sample analysis to reduce random error.

- Begin the sample run, flushing the system with the calibration blank solution between each sample. Analyze the instrument check standard and the calibration blank each ten samples.

8.4.3.1.1.6 Calculations

Calculations of analytical results should be performed in accordance with the manufacturer's instruction for computerized data reduction and in consideration of the following:

- If dilutions were performed, the appropriate factor must be applied to sample values.

- Data must be reported in micrograms per liter (ug/l).

8.4.3.1.1.7 Quality Control

The major QC elements of the analytical procedure are summarized below:

- Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve while the preparation blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

- Calibration Blank: This blank is prepared by diluting 2 milliliters of (1+1) HNO_3 and 10 milliliters of (1+1) HCl to 100 milliliters with deionized, distilled water. Prepare a sufficient quantity to be used to flush the system between standards and samples.

  Analyze the calibration blank at a frequency of 10 percent. The results should be within +/- contract required detection levels (Table 4-1). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

- Reagent Blank (Method/Preparation Blank): This blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

- In addition to the calibration standards, an instrument check standard and an initial calibration verification sample are also required for the analysis.

  - Instrument Check Standard: The standard is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves.

    For continuing calibration verification, analyze an appropriate instrument check standard containing the elements of interest at a frequency of 10 percent. This check standard is used to determine instrument drift. If agreement is not within +/- 10% of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding ten samples reanalyzed.

  - Initial Calibration Verification Sample: This sample consists of a solution obtained from an outside source having known concentration values to be used to verify the calibration standards. This sample should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. If the results are not within +/- 10 percent of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

- A serial dilution interference check must be performed for each group of samples of similar matrix type and concentration or for each 20 samples received, whichever is more frequent.

If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrument detection limit after dilution), an analysis of a 1 to 4 dilution must agree within 10 percent of the original determination. Samples identified as field blanks cannot be used for serial dilution analysis.

If the dilution analysis is not within 10 percent, a chemical or physical interference effect should be suspected and the data must be flagged.

- A matrix spike sample should be analyzed at a frequency of one per 20 samples. An aliquot of sample is spiked as described in the CLP SOW and taken through the entire procedure.

- A duplicate sample should be analyzed at a frequency of one per 20 samples. This sample must be taken through the digestion and analysis.

8.4.3.2 Chloride Analyses

8.4.3.2.1 Method Summary

An acidified sample is titrated with mercuric nitrate in the presence of an indicator. Excess mercury is detected by formation of a blue-violet complex.

8.4.3.2.2 Standards

Standard sodium chloride solution is used to standardize the mercuric nitrate titrant.

8.4.3.2.3 Analysis

Measure an aliquet of sample (generally 50 ml) into an Erlenmeyer flask. Add indicator and adjust pH if necessary. Titrate with standardized mercuric nitrate until a blue-violet color persists.

Titrate a blank consisting of 50 ml deionized water as described above.

8.4.3.2.4 Calculations

$$\text{mg/L Cl} = \frac{(A - B) \times N \times 35{,}450}{\text{mL sample}}$$

where A = mL titrant for sample

B = mL titrant for blank

N = normality of mercuric nitrate titrant

8.4.3.2.5 Quality Control

A blank spike will be analyzed with each group of samples. Results must fall within 80-120%.

An MS/MSD pair will be analyzed for every 20 samples received.

8.4.3.3 Sulfate Analyses

8.4.3.3.1 Method Summary

Sulfate is converted to a barium sulfate suspension. The resulting turbidity is measured by a spectrophotometer and compared against a standard curve.

8.4.3.3.2 Standards

A standard sulfate solution is prepared by dissolving anhydrous sodium sulfate in deionized water. A calibration curve is prepared.

8.4.3.3.3 Analysis

Barium chloride reagent is added to standards and samples. The turbidity for each is measured using a UV/VIS spectrophotometer. The ug sulfate for each sample is determined from a calibration curve.

8.4.3.3.4 Calculations

$$\text{mg/L } SO_4 = \frac{\text{mg } SO_4 \text{ from curve}}{\text{mL sample}}$$

8.4.3.3.5 Quality Control

A blank spike is analyzed with each group of samples. Results must fall within 80-120%.

An MS/MSD pair will be analyzed for every 20 samples received.

8.4.3.4 Alkalinity Analyses

8.4.3.4.1 Method Summary

A sample is titrated to an electrometrically determined end point of 4.5.

8.4.3.4.2 Standards

A prepared sulfuric acid solution is standardized against a THAM standard (tris (hydroxymethyl) - aminomethane).

8.4.3.4.3 Analysis

Add standard acid to an unaltered aliquot of sample until a pH of 4.5 is reached. Record the volume of titrant.

8.4.3.4.4 Calculations

Results are expressed as mg/L CaCO3

$$\text{Alkalinity, mg/L } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{mL sample}}$$

where A = mL standard acid

N = normality of standard acid

8.4.3.4.5 Quality Control

A blank spike is analyzed with each group of samples. Results must fall within 80-120%.

Duplicates are analyzed at a frequency of one for every 10 samples.

8.4.3.5 Carbonate and Bicarbonate Analyses

Carbonate and bicarbonate alkalinity measurements are based on the alkalinity analyses described in Section 8.4.3.4. An additional measurement required is the ml titrant needed to reach a pH of 8.3 (phenolphthalein end point). The following relationships are used:

Alkalinity Relationships*

Result of Titration	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
P = 0	0	T
P < 1/2T	2P	T - 2P
P = 1/2T	2P	0
P > 1/2T	2(T - P)	0
P = T	0	0

*Key: P-phenolphthalein alkalinity; T-total alkalinity.

8.4.3.6 Hardness Analyses

8.4.3.6.1 Method Summary

Calcium and magnesium ions are chelated with EDTA upon addition of an EDTA sodium salt solution. The indicator end point is red in the presence of calcium and magnesium and blue when the cations are chelated.

8.4.3.6.2 Standards

The EDTA titrant is standardized against a standard calcium solution.

8.4.3.6.3 Analysis

An aliquot of sample is measured and neutralized. Buffer, indicator and inhibitor are added. EDTA titrant is added until the blue endpoint is reached.

8.4.3.6.4 Calculations

Results are expressed as mg/L $CaCO_3$.

$$\text{Hardness, mg/L } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml sample}}$$

where: A = mL EDTA titrant

N - normality of EDTA titrant

8.4.3.6.5 Quality Control

A blank spike is analyzed with each group of samples. Results must fall within 80-120%.

Duplicates are analyzed at a frequency of one for every 10 samples.

8.4.3.7 Total Dissolved Solids (TDS) Analysis

8.4.3.7.1 Method Summary and Analysis

An aliquot of well-mixed sample is filtered through a glass fiber filter. The filtrate is transferred to a prepared beaker, evaporated and dried at 180°C.

8.4.3.7.2 Calculations

$$\text{TDS, mg/L} = \frac{(A - B) \times 1{,}000}{\text{mL sample}}$$

where: A = weight of dried residue and beaker

B = weight of beaker

8.4.3.7.3 Quality Control

A blank spike is analyzed with each group of samples. Results must fall within 80-120%.

Duplicates are analyzed at a frequency of one for every 10 samples.

8.4.3.8 pH Analyses

8.4.3.8.1 Method Summary

The pH of a sample is determined electrometrically. pH meters read directly in pH units.

8.4.3.8.2 Analysis

The pH meter is calibrated before each use with two buffers (no more than 3 pH units apart) that bracket the sample pH. The pH of the samples are then measured.

8.4.3.8.3 Quality Control

The pH meter calibration is checked after every 10 samples.

Duplicates are analyzed at a frequency of one for every 10 samples.

8.4.3.9 TOC Analyses

8.4.3.9.1 Organic carbon is converted to carbon dioxide by either combustion or wet chemical oxidation. The CO_2 formed is measured directly by an infrared detector.

8.4.3.9.2 Standards

The instrument is calibrated using a potassium hydrogen phthalate standard.

8.4.3.9.3 Analysis

An aliquot of acidified sample is purged to remove inorganic carbon. The purged sample is then injected into the calibrated instrument. The instrument reads directly in mg/L TOC.

8.4.3.9.4 Quality Control

A blank spike is analyzed with each group of samples. Results must fall within 80-120%.

An MS/MSD pair will be analyzed for every 20 samples received.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

The following procedures summarize the Standard Operating Procedures (SOPs) routinely used by the ITAS and engineering groups for data reduction, validation, and reporting.

9.1 Analytical Laboratory Data

All laboratory results and QA data will be reviewed by the appropriate group leader and QA officer. For volatile organic compounds and metals analyses, CLP forms will be submitted only. For anions, hardness, TDS, alkalinity, pH, TOC, and TPH analyses, a regular certificate of analysis will be submitted and QA/QC data will be reported with the analytical results. These QA data will be used to validate data integrity.

Laboratory calculations and data reduction are independently checked in accordance with Section 10.2.1 of the IT Analytical Services Quality Assurance Manual. This process must be satisfactorily completed for the data to be considered valid.

9.1.1 Data Verification

- Calculate QC data before completing other calculations and before reporting data.
- Calculate analyses results and complete data sheets; sign and date each page.
- Request that another analyst or supervisor approve the notebook or laboratory data sheets by formal checking.
- Record data on project data summary sheets as necessary; initial and date forms.
- Check other parameters for relative concentration values (alert supervisor, if necessary).

- File instrument charts (e.g., metals) in appropriate data files; enter information required on form.

- Enter QC data on appropriate forms and charts.

9.1.2 Report Preparation

- Review data on project data sheets and previous similar project data, if available.

- Check laboratory data sheets and non-conformance memos for comments regarding sample analyses.

- Review detection limits and report summarized data with appropriate significant figures and units.

- Submit data for report preparation.

- Verify typed data for formal checking.

- Discuss results with project manager prior to submittal of report.

- Report results externally.

- Transmit appropriate records to laboratory project files.

9.1.3 Quality Assurance Review

The QA Officer shall review testing results prior to external distribution. This review shall include:

- Compare analyses performed to the proposed testing record.

- Review results for reasonableness.

9.2 Engineering Analysis and Calculations

Analysis activities performed in the office shall be completely documented and the resulting documentation formally checked by peers.

9.2.1 Numerical Analysis Procedures

Analysis activities shall be performed in a planned and controlled manner. Performance responsibility rests with the project manager. Prior to initiating the activities, the manager shall discuss the scope of the work, contractual and regulatory requirements, and applicable QA/QC procedures with assigned personnel. At the request of the project manager, this will be done by the QA group.

To provide evidence of satisfactory work performance and the basis for information transmittal, analyses and their results shall be completely documented. Documentation may include calculations, computer programs, logs, drawings, and tables.

9.2.1.1 Calculations

Calculations shall be legible and in a form suitable for reproduction, filing, and retrieval. Documentation shall be sufficient to permit a technically qualified individual to review and understand the calculations and verify the results.

Calculations shall be performed on standard calculation paper whenever possible. All calculation pages shall be individually identified, with the exception of large computer output. Calculation paper provides spaces for the originator's name and date of work, the checker's name and date, calculation subject, project number, and page number. All of this information shall be completed for each page. For extra pages, such as large graphs, this information shall also be included.

Calculations should, as appropriate, include a statement of calculation intent, description of methodology used, assumptions and their justification, input data

equation references, numerical calculations including units, and results. Input data may include:

- Regulatory requirements.
- Performance and operational requirements under various conditions.
- Material, geological, environmental, and geotechnical requirements.
- Results of field and laboratory testing or calculations.
- Information obtained from external personnel or literature and site data surveys.

Computer printout that becomes an integral part of the calculations shall be referenced in the calculations by run number or other unique means of identification. At the end of the calculations, the results should be summarized if this will provide clarity.

9.2.1.2 Computer Programs

Computer programs used for analysis shall be documented and verified in accordance with applicable requirements. Computer output shall be dated and clearly identified as to contents. Large sets of output shall be labeled with project name and number, program used, analysis title, and the user's name.

9.2.1.3 Logs, Drawings, and Tables

The results of analysis activities may be presented in logs, drawings, and tables of various forms. The format of logs and tables shall be governed by the information to be presented. Drawings shall be uniquely identified by drawing or figure number and appropriate title. Standardized symbols and nationally accepted drafting

standards shall be used. References to other drawings and sources of information shall be provided, as necessary.

Drawings shall be signed and dated by the draftsperson performing the work and the responsible member of the project group who has checked the drawing.

Revisions shall be noted on the drawing original with a revision number and a brief note describing each revision. The note shall be signed and dated by the draftsperson performing the work and the responsible member of the project group who has checked the revision.

Indication of final drawing and revision approval shall be through signing and dating of the drawings by the project manager or a member of the project staff delegated this responsibility by the project manager. Approval does not indicate checking but only approval for issue.

9.2.2 Analysis Verification

Calculations, computer program input, logs, drawings, and tables shall be formally checked using the process outlined in the following sections.

9.2.2.1 Calculations

For calculations, assignments for checking shall be made or approved by the project manager. Verification shall be performed by an individual(s) other than the persons who performed the original work or specified the method or input parameters to be used. The individual(s) selected shall have technical expertise in the calculation subject.

It is emphasized that a numerical check is not sufficient. The checker is responsible for every item on every sheet--including the completion of the title block and page numbers. To properly check calculations:

- The originator supplies the designated checker with a copy of the calculations. Originals should not leave the originator's possession until they are ready for final checker signing.
- The checker marks the calculation copy with a yellow marker for all items he approves.
- If the checker disagrees, for any reason, the checker crosses through the item with a red marker and writes the recommended correction or comment above it.
- The checker returns the checkprints to the originator who, in turn, reviews all recommended changes. If a disagreement exists, the originator adds comments to the checkprints using a third color and then confers with the checker until all differences are resolved.
- The originator corrects, or "scrubs," the calculation originals so they agree with the checkprints. A one-to-one correspondence between the originals and checkprints must exist.
- The originator gives the originals and checkprints to the checker who compares them to verify agreed-to corrections have been made.
- When the checker is satisfied, he signs and dates the originals.

9.2.2.2 Computer Program Input

Computer input shall be formally checked using the process outlined in Section 9.2.2.1 above. A single exception to this is that the checking may be performed on the input originals. The verification shall include a conceptual review of the program itself based on the problem being solved, a review of the computer model employed, a check that the program has been verified, and a formal check of the input data.

9.2.2.3 Drawings

Drawings shall be checked like calculations (Section 9.2.2.1) using yellow and red markers. Checkprints of the same drawing shall be marked CP1, CP2, etc., to show the progression of the checking process.

If a drawing is revised, the entire checking process shall be repeated for the revised area only. A new checkprint shall be prepared. Under no circumstances shall revisions be made without the formal checking procedure.

9.2.2.4 Logs and Tables

Final subsurface logs shall be verified by the responsible member of the project group. The verification shall provide that changes from the original field representative's logs to the final log sheets are consistent with the results of other investigations. The final log sheet shall be checked in the same manner as all calculations or drawings, with the checker signing and dating all checkprints.

In addition, all final tables presenting information, data, or the results of analyses shall be checked using the process in Section 9.2.2.1. Checkprints of the same table shall be marked CP1, CP2, etc., to show progression of the checking process.

10.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

10.1 Quality Assurance Review of Reports, Plans, and Specifications

Prior to issuance of a final report, it shall be reviewed by knowledgeable members of the project staff and the project manager, or designated representative. This review shall address whether:

- The report satisfies the scope of work, client requirements, and pertinent regulatory requirements.
- Assumptions are clearly stated, justified, and documented.
- A reference is cited for any information utilized in report preparation that was originated outside the project.
- The report correctly and accurately presents the results obtained by the project work.
- The tables and figures presented in the report are prepared, checked, and approved.
- The report figures are signed and dated by the appropriate members of the project staff and project management.
- The bases for the recommendations and conclusions presented in the report are clearly documented.
- The typed report has been proofread and punctuation, grammar, capitalization, and spelling are correct.

In addition to review by the project group, specific reports may undergo a peer review process. Peer review should be considered for all reports and is recommended for those which present state-of-the-art technical activities, regulatory review, or the results of projects having great potential for litigation. The peer review process is intended to complement the verification of numerical analyses and data

processing. While verification provides review and confirmation of largely definitive work, peer review provides evaluations and assessment of interpretations, judgments, and decisions made.

Peer reviews shall be coordinated by the Project Manager. The reviews shall be performed by personnel not involved with the activities presented in the report but who have technical expertise at least equivalent to those performing the work. Reviews should address the following, as appropriate:

- Were the work plans and procedures, as developed, sufficient?
- Have procedures been correctly used?
- Did the procedures used result in obtaining data objectives of the project and have the objectives been properly translated from applicable contractual requirements, industry standards, and federal/state/local regulations?
- Have sufficient data of adequate quality been collected to reach conclusions which can be justified and verified?
- Are assumptions, interpretations, judgments, or decisions supported by the data and are they defendable?
- What are the effects of variations upon results?
- Is documentation sufficient to verify the validity and reproducibility of report results?

Peer reviews shall be documented. It is recommended that the reviewer document the following:

- Date(s) of review
- Participants
  - Reviewers
  - Project personnel
  - Any QA personnel

- Activities reviewed including interpretations, decisions and judgments.
- Statement of agreement or disagreement with interpretations, decisions, and judgments; including the means for resolving disagreements.
- Recommendations for changes, the extension of existing activities, or the establishment of additional studies.
- If questions remain unresolved beyond the end of the review, the means for closing the questions.

Review reports shall be transmitted to project management and appropriate QA personnel.

10.2 Quality Control Checks and Procedures

All QC operations for metals and volatile organics must be performed in accordance with the respective CLP SOW. These operations were discussed in Section 8. The SOW revision current at the time of sample analyses will be used. Corrective actions will be as described by the CLP protocol.

QC operations for total petroleum hydrocarbons, anions, alkalinity, hardness, TDS, pH an TOC are described in Section 8. MS/MSD pairs will be analyzed at a frequency of one per 20 samples of a specific matrix. If the MS/MSD are not found to be appropriate, duplicates will be analyzed at a frequency of one per 10 samples. The QA objectives are as listed in Table 4-1. If these objectives are not met, reanalyses will be performed to confirm a matrix effect.

10.3 Field Quality Control

Field activities are the responsibility of the Project Manager and Site Coordinator. Prior to initiating field work, the Project Manager shall discuss the scope of work, contractual and regulatory requirements, and applicable QA/QC procedures

with assigned personnel. At the request of the Project Manager, this may be done by QA personnel or senior members of the project staff.

Once in the field, field personnel are responsible for all daily QC activities. Included in this responsibility shall be the supervision of subcontractors, and implementation of the RI/FS SOPs in Appendix A.

Results of the field investigation shall be completely documented. Whenever possible, information shall be recorded on a standardized form, such as those in the RI/FS SOPs in Appendix A. Documentation shall include a daily log of project activities and the appropriate subsurface logs, test and survey data forms, monitoring/sampling equipment installation records, photographs, and field collection and sampling custody forms, as required in the RI/FS SOPs in Appendix A.

11.0 QUALITY ASSURANCE AUDIT

To verify compliance with specific project QA/QC Program requirements, the IT QA group shall perform a planned and documented audit of the project activities. This audit shall consist, as appropriate, of an evaluation of work areas and activities, and a review of project documentation. The audit shall be performed in accordance with written checklists by trained members of the QA group and, as appropriate, technical specialists. Audit results shall be formally documented and sent to project management.

The audit may include, but not be limited to, the following:

- Field operations records.
- Laboratory testing and records.
- Equipment calibration and records.
- Identification and control of samples.
- Numerical analyses.
- Computer program documentation and verification.
- Transmittal of information.
- Record control and retention.

The planned audit for this project will, as appropriate, cover the final reports. Auditing will be performed in accordance with applicable requirements of Section 1.8 of the IT Engineering Services Quality Assurance Manual.

An individual audit plan shall be developed to provide a basis for each audit. This plan shall identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the schedule. The plan shall be consistent with the project scope, schedule and requirements.

A report audit may examine, as appropriate, the documentation and verification of field and laboratory data and results; performance, documentation, and verification of analyses; documentation and verification of computer programs; preparation and verification of drawings, logs, and tables; content, consistency, and conclusions of the report; compliance with project, regulatory, and project requirements; and maintenance and filling of project records.

The records of field operations shall be reviewed to verify that field-related activities were performed in accordance with appropriate project procedures. Items reviewed shall include, but not be limited to, the calibration records of field equipment; daily field activity logs; photographs; and data, logs, and checkprints resulting from the field operations.

The auditing laboratory testing records shall include, but not be limited to, the originals and checkprints of laboratory data sheets, originals and checkprints of data presentations prepared by the laboratory staff, and laboratory test scheduling records for the project.

Auditing of analyses shall include a complete review of calculations, computer input, sketches, charts, tables, and their associated checkprints that were prepared by

the project group. These items shall be reviewed to verify conformance to project requirements.

The report preparation process shall be reviewed to verify that:

- The report correctly and accurately presents the results obtained by the work.
- All information presented in the report is substantiated by project work.
- The tables and figures presented in the report are prepared and checked.
- The report satisfies the scope of work, project requirements, and any pertinent regulatory requirements.

A report audit shall also, as appropriate, review the maintenance and control of project documents to verify that applicable procedures have been implemented.

A report audit shall be performed prior to issuance of a final report. The issuance of the final submittal shall be postponed if the QA group determines that the work does not meet requirements. If the project schedule demands issuance of a report prior to audit, it may be issued "preliminary" or "draft" or WPAFB may be formally notified that an internal audit is in progress and that the submittal will be considered final only after completion of the audit.

Checklists shall be prepared by the auditors and used to conduct all audits. They shall be developed to accomplish the review of necessary items and to document the results of the audit.

During an audit and upon its completion, the auditors will discuss the findings with the individuals audited and cite corrective actions to be initiated. Minor

administrative findings which can be resolved to the satisfaction of the auditors during an audit are not required to be cited as items requiring corrective action. All findings that are not resolved during the course of the audit and findings affecting the overall quality of the project, regardless of when they are resolved, shall be noted on the audit checklist.

Appendix C contains results of the most recent CLP performance evaluation and audit for ITAS laboratory in Cincinnati, Ohio.

12.0 PREVENTIVE MAINTENANCE

Preventive maintenance is an organized program, within ITAS, of action (such as equipment cleaning, lubricating, reconditioning, adjustment, and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. An adequate preventive maintenance program increases reliability of a measurement system. A preventive maintenance program considers the following:

- Instruments, equipment, and parts thereof that are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance.
- Spare parts that should be available within the laboratory to minimize downtime.
- Frequency that maintenance is required.

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used within an ITAS laboratory; therefore, this manual does not designate specific practices for instruments and equipment. Each ITAS laboratory will prepare a preventive maintenance program which meets the guidelines presented in this section.

Within a laboratory, the laboratory director is responsible for preparation and documentation of the program. Group leaders shall implement the program and the QC Coordinator shall review implementation to verify compliance.

The ITAS preventive maintenance program shall include the following:

- A listing of the instruments and equipment that are included in the program.
- The frequency of maintenance considering manufacture's recommendations and/or previous experience with equipment. The listing and maintenance frequency should be provided on a schedule. Frequency shall be stated in terms of monthly, quarterly, etc.
- For each instrument in the program provide:
  - A list of spare parts maintained by the laboratory
  - External services contracts
  - Items to be checked and/or services during maintenance (if external services are not provided or if not stated in manufacture's instrument manuals).

The ITAS Preventive Maintenance schedule is included in Appendix B. A summary is provided in Table 12-1. Preventive maintenance should be documented as discussed below and the records stored in accordance with Section 12.2. of the IT Analytical Services Quality Assurance Manual. The master schedule should be kept at the beginning of the maintenance records. Behind the schedule, a separate file should be maintained for each instrument. The instrument file should include:

- Spare parts list.
- External service contracts.
- Checklist of items to be serviced and directions for maintenance or manufacturer's instrument manuals.
- Record of periodic maintenance.

The record of maintenance is documented in bound notebooks which are kept with the corresponding laboratory instrument.

TABLE 12-1. PREVENTIVE MAINTENANCE SUMMARY

Instrument	Item checked/serviced	Frequency
Balances	External service calibration	Annually
Deionized/Milli-Q Water	Conductivity check Cartridges changed	Daily As needed
Refrigerators	Temperature checked and logged	Daily
pH meter	Electrode cleaned	Monthly or as needed
	Outside of meter wiped down	Weekly
UV/Vis spectrophotometer	Instrument response checked with commercially-purchased solutions	Monthly
	Wavelength checked with didymium filter	Monthly
	Outside of instrument wiped down	Weekly
Conductivity meter	Conductivity cell cleaned	Quarterly
	Replatinization	As needed
TOX	Inlet and outlet tubes checked for residue buildup	Daily
	Inlet and outlet tubes cleaned or replaced	As needed
	Cell electrodes checked for residue buildup	Daily
	Cell electrodes cleaned or replaced	As needed
	Electrolyte replenished	As needed
	Outside of instrument wiped down	Weekly
TOC	Septa replaced	As needed
	Glass wool in boat replaced	Daily
	Reagent in reaction vessel replenished	Daily
	Tin and copper scrubbers repacked	As needed
	Pump tubing replaced	As needed
	Lithium hydroxide scrubber repacked	As needed
	Cupric oxide furnace tube repacked	As needed
	Outside of instrument wiped down	Weekly

(continued)

TABLE 12-1 (continued)

Instrument	Item checked/serviced	Frequency
Ion chromato-graph - Column	Outside of instrument wiped down	Weekly
	Cleaned with 0.1 M Na_2CO_3, stored in 0.1 M NaOH	When not in use
	Cleaned with methanol	As needed
	Column replaced	As needed
	Fiber suppressor replaced	As needed
- Pump	Seals changed Oil added	Semiannually Monthly
- Autosampler	Plunger tip and tubing changed	Annually
AA spectro-photometer	Burner head checked	Daily
	Burner head cleaned	As needed
	Flow chamber checked and rinsed	Daily
	Flow chamber cleaned	As needed
	Nebulizer checked and rinsed	Daily
	Nebulizer cleaned	As needed
	Drain tube filled	Daily
	D_2 lamp changed	Semiannually or as needed
- Furnace	Graphite tube checked	Daily
	Contract rings checked	Daily
	Contact rings cleaned or replaced	As needed
	Quartz windows checked	Daily
	Quartz windows cleaned or replaced	As needed
ICP spectro-photometer	Pump tubing replaced	Daily
	Capillary tubing checked	Daily
	Capillary tubing replaced	As needed

(continued)

TABLE 12-1 (continued)

Instrument	Item checked/serviced	Frequency
	Torch quartz tube checked	Daily
	Torch quartz tube cleaned or replaced	As needed
	Torch injector tube checked	As needed
	Torch injector tube cleaned or replaced	As needed
	Nebulizer insert cleaned or replaced	As needed
	Drain bottle emptied	As needed
	Nebulizer insert checked	As needed
	Pump oil changed	Semiannually
GC	Column packing replaced Detector cleaned Glass wool plug changed Insert cleaned	Determined by analyst so that the calibration is within required specifications
	EC (Ni-63) wipe test	Semiannually or per NRC requirements
	Septa replaced	After each production sequence
GC/MS	Clean ion source	As needed
	Replace filament	As needed
	Clean quadrupole rods	As needed
	Replace resistor or electron multiplier	As needed
	Replace o-ring on analyzer assembly flange; inspect RF voltage feed-throughs for damage	As needed
	Replace o-ring on magnet well flange or front cover port	As needed
	Check air filter for obstruction	Monthly
	Change air filter	Quarterly

(continued)

TABLE 12-1 (continued)

Instrument	Item checked/serviced	Frequency
	Check cooling fans	Monthly
	Clean cooling fans	Quarterly
	Inspect signal cable on digital I/O PCBA	Quarterly
	Inspect oscilloscope monitor cable on digital I/O PCBA	Quarterly
	Inspect all PCBAs, interior of module	Quarterly
	Thoroughly clean all PCBAs, interior of module	Annually
	Clean turbomolecular pump fan	Quarterly
	Purge rotary vane vacuum pump	Monthly
	Change rotary vane vacuum pump oil	Quarterly
	Change turbomolecular pump oil	Quarterly
	Regenerate air filter/drier desiccant	Semiannually or as needed
	Inspect solenoid-operated valves	Quarterly
	Bake out vacuum manifold	Quarterly
	Inspect Tygon tubing and connectors	Quarterly
	Check Pirani gauges	Quarterly
	Replace ferrules (interface over and transfer line)	Quarterly
	Clean disk drive cooling fan	Quarterly
	Clean power-supply cooling fan	Quarterly
	Clean magnetic tape drive	Quarterly
	Clean and lubricate auto sampler	Quarterly
	Check ethylene glycol/water (50/50) level in Neslab coolers	Monthly

13.0 DATA ASSESSMENT PROCEDURES

As part of the analytical QC Program, the laboratory will apply the precision and accuracy criteria specified in the U.S. EPA CLP protocols previously cited for metals and volatiles (see Table 4-1). For total petroleum hydrocarbons and other inorganic parameters, the accuracy and precision limits in Table 4-1 will be used. When the analysis of a sample set is completed, the QC data generated are reviewed and evaluated to validate the data set. The review is based on the following criteria:

- Reagent/Method Blank Evaluation - The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis of future samples halted until the system can be brought under control. A high background is defined as a background value sufficient to result in a difference in the sample values, if not corrected, greater than or equal to the smallest significant digit known to be true. A method blank should contain no greater than two times the parameter detection limit for most parameters. (1,2,3)[1]

- Field Blank Evaluation - Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered (i.e., a value sufficient to result in a difference in the sample values, if not corrected, greater than or equal to the smallest significant digit known to be true), the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. Ambient air in the laboratory and reagents should be checked as possible sources of contamination. High field blank readings may also be due to contaminated sample bottles or cross contamination due to sample leakage and poorly sealed sample containers. (1,2,3)[1]

- Standard Calibration Curve and Response Factor Evaluation - The calibration curve or midpoint calibration standard (check standard) is the evaluated ability to determine curve linearity through its full range and

that sample values are within the range defined by the low and high standards. If the curve is not linear, sample values must be corrected for nonlinearity before deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards. In addition, if average response factors are used to calculate sample concentrations, these factors will be verified on a daily basis. Verification of calibration curves and response factors is accomplished when the evaluated response for any parameter varies from the calibrated response by less then ranges given in the applicable test methods listed in the EPA CLP protocols. (1,2,3)[1]

- Duplicate Sample Evaluation - Duplicate sample analysis is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the RPD. If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question. (2)[1]

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where D_1 = first sample value
D_2 = second sample value (duplicate)

- QC Standard Evaluation - The results of check standard analyses are compared with the true values and the percent recovery of the check standard is calculated. If correction is required (excessive or inadequate percent recovery), the check standard should be reanalyzed to demonstrate that the corrective action has been successful. (1,2,3)[1]

- Surrogate Standard Analyses - Surrogate standard determinations should be performed on all samples and blanks for GC/MS analyses. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction and analysis of samples. Recoveries should meet U.S. EPA CLP acceptance criteria as presented in Table 4-2. If acceptance criteria are not met, corrective action is taken to correct the problem and the affected sample is reanalyzed. (1)1

- Matrix Spike/Matrix Spike Duplicate Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. If the accuracy value exceed the control limit for the given parameter (Tables 4-1 and 4-2), the appropriate laboratory personnel are notified and corrective action is

taken. The RPD between duplicate matrix spikes is also assessed to evaluate the precision. The acceptance criteria in Table 4-2 must be achieved or corrective action must be taken before the affected samples are reanalyzed. (1,2,3)[1]

$$\% \ Recovery = \frac{(SSR - SR)}{SA} \times 100$$

where SSR = spiked sample result
SR = sample result
SA = spike added

- Trip Blank Evaluation - Trip blanks are used to evaluate cross-contamination of volatiles during shipment.

[1] Criteria are referenced as applicable to:

1 - Volatile organic compounds. (TCL)
2 - Metals (TCL)
3 - Total Fuel Hydrocarbons

14.0 NONCONFORMANCE/CORRECTIVE ACTION

Nonconforming items and activities are those which do not meet the project requirements, procurement document criteria, or approved work procedures. Nonconformances may be detected and identified by:

- Project Staff - During the performance of field investigation and testing, supervision of subcontractors, and preparation and verification of numerical analyses
- Laboratory Staffs - During the preparation for and performance of laboratory testing, calibration of equipment, and QC activities
- QA Staff - During the performance of audits.

Each nonconformance affecting quality shall be documented by the personnel identifying or originating it. For this purpose, a variance log (Figure 14-1), testing procedure record, notice of equipment calibration failure, results of laboratory analysis QC tests, audit report, internal memorandum, or letter shall be used as appropriate. Documentation shall, when necessary, include:

- Identification of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance
- Any required approval signatures
- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted.

FIGURE 14-1

VARIANCE LOG

PROJECT NUMBER _____ PAGE _____ OF _____

PROJECT NAME _____

DATE	VARIANCE GRANTED AND APPLICABLE DOCUMENT	RESPONSIBLE INDIVIDUAL

Documentation shall be made available to project, laboratory, and/or QA management, and , if applicable, subcontractor management. It is the responsibility of the Project Manager, Laboratory Director, and/or Corporate Director of QA to then notify appropriate personnel of the nonconformance. In addition, the Project Manager will notify Battelle EMO and WPAFB of significant nonconformances which could impact the results of the work and indicate the corrective action taken or planned.

Completion of corrective actions for significant nonconformances should be verified by the QA group as part of future auditing activities.

Any significant recurring nonconformance should be evaluated by project, laboratory, and/or QA personnel to determine its cause and appropriate changes instituted in project requirements and procedures to prevent future recurrence. When such an evaluation is performed, the results shall be documented.

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

There are many forms of work quality reporting to various levels of management. Adequate provisions have been made within this QA Project Plan for the reporting of quality-related considerations. Quality-related reports submitted to management include:

- The reporting by project personnel of nonconformances to project management so that corrective action can be taken. These reports will be submitted upon identification of the nonconforming activity.
- Reporting of the QC coordinator to laboratory management concerning data accuracy and precision achieved through QC sample analyses and analyses of performance evaluation samples. These reports are typically in the form of control charts, which are prepared on a regular basis and submitted monthly.
- Reporting of the QC Coordinator to laboratory management of nonconformances observed during internal laboratory system audits. A schedule for internal audits is presented on Table 15-1.
- System audits conducted for work assignments by the engineering QA officer. These audits are conducted frequently during and following major work activities.
- System audits of overall project performance conducted by the Corporate Director of QA.

Following completion of an audit, the auditors shall prepare and submit an audit report to the IT Vice President of Engineering, program director, project manager, and Corporate Director of QA. This report shall serve to notify management of audit results. The report may also be sent to individuals contacted during the audit and the management of any affected subcontractor.

TABLE 15-1
ITAS - CINCINNATI LABORATORY INTERNAL AUDIT SCHEDULE

January	Gas Chromatography/Mass Spectrometry (GC/MS)
February	Sample Control
March	None
April	Inorganics
May	Organic Extraction
June	Gas Chromatography (GC)
July	GC/MS
August	Sample Control
September	None
October	Inorganics
November	Organic Extraction
December	GC

The report shall be prepared as soon as possible (within 30 days) after the audit and contain, as appropriate:

- Date(s) of the audit.
- Identification of audit participants.
- Identification of activities audited.
- Audit results.
- Description of items requiring corrective action and, if possible, the means of correction.
- Due date for completion of corrective actions and/or audit response.
- Means for audit response (in writing).

If corrective action is required in the audit report, the corrective action shall be undertaken and completed on schedule unless sufficient evidence can be provided through management receiving the audit report to prove that the action is unnecessary. If required, the Corporate Director of QA is empowered to stop work on the project pending resolution.

The individuals audited shall respond in writing to the audit report. The response shall clearly state the corrective action taken or planned. If all corrective actions have not been completed prior to issuance of the audit response, a scheduled date for completion shall be provided. It is noted that all requests for corrective action must be addressed to the satisfaction of the Corporate Director of QA.

Completion of corrective action shall be verified by the auditors through written communication, reaudit, or other appropriate means. After acceptance and verification

of corrective actions, an audit closure shall be issued by the auditors to the same individuals receiving the audit report.

REFERENCES CITED

Weston, Inc. September 1985. Final Report Phase II-Stage 1 Study. Prepared for Wright-Patterson Air Force Base.

Weston, Inc. July 1989. Stage 2 Report-Volumes I, II, and III, Technical Report and Appendices. Prepared for Wright-Patterson Air Force Base.

Engineering-Science, Inc., 1990a. Project Work Plan for Remedial Investigations and Feasibility Studies at Wright Patterson Air Force Base, Ohio, Volume 1, Project Program Description for 39 IRP Sites.

Engineering-Science, Inc., 1990b. Project Work Plan for Remedial Investigations and Feasibility Studies at Wright Patterson Air Force Base, Ohio, Volume 2, Quality Assurance Project Plan.

APPENDIX A

DRAFT STANDARD OPERATING PROCEDURES AND AMENDMENTS

THIS APPENDIX IS PRESENTED IN VOLUME 3A

APPENDIX B

STANDARD OPERATING PROCEDURES FOR IT ANALYTICAL SERVICES CINCINNATI, OHIO

INTERNATIONAL TECHNOLOGY CORPORATION

SOP NO.: 8901-ICP-01
DATE INITIATED: 04/10/89
REVISION NO.: 0
DATE REVISED:

STANDARD OPERATING PROCEDURE

FOR ANALYSIS OF METALS

BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES DIVISION

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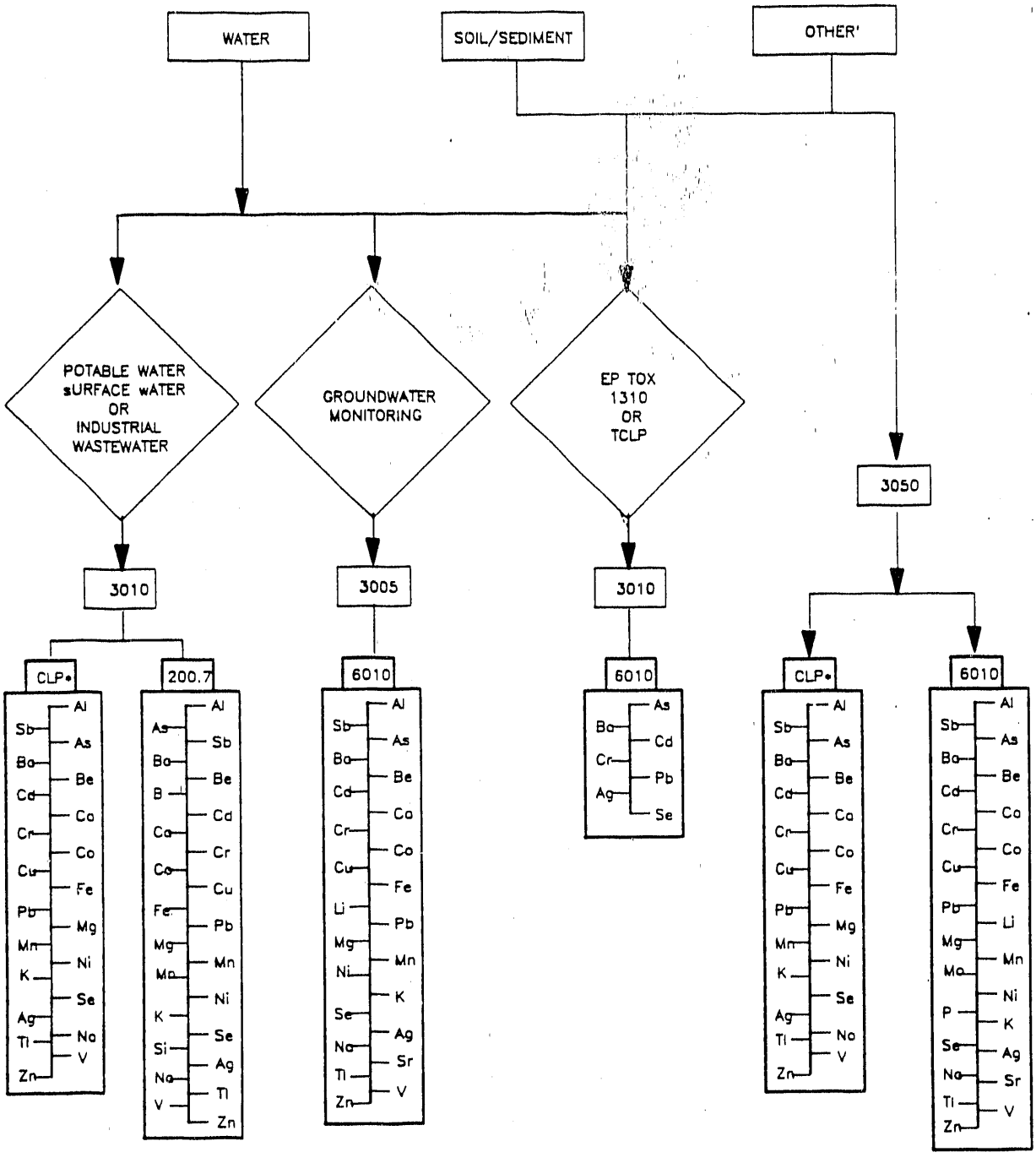
TECHNICAL OVERVIEW

Inductively coupled plasma atomic emission spectroscopy is based on the element specific emission spectra produced by introduction of a sample into a radio-frequency inductively coupled argon plasma. Currently available instrumentation is usually classified as either simultaneous or sequential. This refers to the detection of the emission lines and is unrelated to the plasma generation. Simultaneous ICP instruments are equipped with dedicated photomultiplier tubes for each element which is to be detected. Sequential ICP instruments are equipped with a movable grating which can be programmed to scan different wavelengths for the elements of interest. Simultaneous ICP instruments are inherently more stable than sequential instruments but the addition of new elements to the scan requires hardware changes. Sequential instruments can be programmed for new elements fairly easily but are subject to drift and changes in sensitivity due to the physical movement of the focusing elements.

The base method selected in the Standard Operating Procedure is the USEPA Contract Laboratory Program Statement of Work No. 787. This procedure is used for remedial investigation/site feasibility studies and ongoing monitoring of clean-up activities. In addition, because this method is based on EPA Method 200.7, it is appropriate for the examination of ground and surface waters, domestic and industrial waste effluents, and treatment process samples. EPA Method 200.7 is approved for water and waste monitoring under the Safe Drinking Water Act (SDWA) and the National Pollutant Discharge Elimination System (NPDES). A list of Target Analytes and Contract Required Detection Limits appears in Table 1.

Appendix A contains the Addendum to the base method for EPA Method SW-846 6010. It contains additional QC as per the method and extends the analysis to include Lithium, Molybdenum, Phosphorous, and Strontium. Appendix B contains the Addendum to the base method for EPA Method 200.7. It extends the analysis to include Boron, Molybdenum and Silicon and includes different QC control limits.

METHOD SELECTION



•NOTE: Compare required detection limit with IDL for each element to decide between ICP & GFAA

STANDARD OPERATING PROCEDURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure is to describe in detail the methodologies used, by the laboratories within the International Technology Analytical Services division in the performance of the analysis of metals in various media by Inductively Coupled Plasma Atomic Emission Spectroscopy.

2.0 SCOPE AND APPLICATION

2.1 Inductively coupled plasma atomic emission spectroscopy (ICP) determines elements including metals in solution. The method is applicable to a large number of metals and wastes. All matrices, including groundwater, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

2.2 Elements for which this SOP is applicable are listed in Table 2. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. The data shown in Table 2 provide estimated instrumental detection limits for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2.3 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L.

2.4 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved

solids content of the samples, appropriate steps must be taken to correct for potential interference effects.

2.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

3.0 SUMMARY OF METHOD

The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical lines, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 6.1 (and tests for their presence as described in 6.2) should also be recognized and appropriate corrections made.

4.0 SAMPLE HANDLING AND PRESERVATION

Sample holding times, digestion procedure and suggested collection volumes are listed in Table 3. The sample volumes required depend upon the number of different digestion procedures necessary for analysis. The indicated volumes in Table 3 refer to that required for the individual digestion procedures and recommended sample collection volumes.

In the determination of trace metals, containers can introduce either positive or negative errors in the measurement of trace metals by (a) contributing contaminants through leaching or surface desorption, and (b) depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis require particular attention. The following cleaning treatment sequence has been determined to be adequate to minimize contamination in the sample bottle, whether borosilicate glass, linear polyethylene, polypropylene, or Teflon: detergent, tap water, 1:1 nitric acid, Type II water, 1:1 hydrochloric acid and Type II water. *NOTE:* Chromic acid should not be used to clean glassware, especially if chromium is to be included in the analytical scheme. Commercial, non-chromate products (e.g., Nochromix) may be used in place of chromic acid if adequate cleaning is documented by an analytical quality control program. (Chromic acid should also not be used with plastic bottles.)

5.0 DEFINITIONS

5.1 Dissolved -- Those elements which will pass through a 0.45 μm membrane filter.

5.2 Suspended -- Those elements which are retained by a 0.45 μm membrane filter.

5.3 Total -- The concentration determined on an unfiltered sample following vigorous digestion.

5.4 Instrument detection limit (IDL) is determined quarterly by analyzing a standard three to five times the estimated instrument detection limit for seven replicate measurements on three non-consecutive days. The average standard deviation multiplied by three is considered to be the IDL.

5.5 Sensitivity -- The slope of the analytical curve (i.e., functional relationship between emission intensity and concentration).

5.6 Instrument check standard -- A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. This standard should be prepared from a different source than the calibration standards.

5.7 Interference check sample -- A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

5.8 Quality control sample -- A solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

5.9 Calibration standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

5.10 Linear dynamic range -- The concentration range over which the analytical curve remains linear. This is to be established and updated or verified quarterly.

5.11 Reagent blank -- A volume of deionized, distilled water containing the same acid matrix as the prepared/digested samples carried through the entire analytical scheme (see 9.6.2).

5.12 Calibration blank -- A volume of deionized, distilled water containing the same acid concentration as the calibration standards (see 9.6.1).

5.13 Method of standard addition (post digestion spike) -- The single standard addition technique involves the addition of a known amount of standard to the digested sample. A percent recovery (%R) must be generated. If the %R is <75 or >125, multiple standard additions (usually 3 points) may be used to quantitate.

6.0 INTERFERENCES

6.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

6.1.1 Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer inter-element correction (IEC) of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array.

Listed in Table 4 are some interference effects for the recommended wavelengths given in Table 2. The data in Table 4 are intended for use only as a rudimentary guide for the indication of potential spectral interferences.

Linear relations between concentration and intensity for the analytes and the interferents must be established prior to determining IECs. The interference information, which was collected at the Ames Laboratory*, is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of the interferent element.

The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 4, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 4, and that the interference effects must be evaluated for each individual system. Only those interferents listed were investigated. The blank spaces in Table 4 indicate that measurable interferences were not observed from the interferent concentrations listed. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2-5% of the peaks generated by the analyte concentrations also listed in Table 4.

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that

*Ames Laboratory, USDOE, Iowa State University, Ames, Iowa 50011.

second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

6.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. Also, the use of matrix matching between standards and samples can be useful. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques.

Another problem which can occur from high dissolved solids is salt build-up at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

6.1.3 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

6.2 The following test must be performed prior to reporting concentration data for analyte elements: for each group of samples of a similar matrix and concentration (i.e., low, medium), for each Case of samples, for each 20 samples received, or for samples received over a 14 calendar day period, whichever is more frequent.

6.2.1 Serial dilution -- If the analyte concentration is sufficiently high (minimally a factor of 50 above the instrument detection limit after dilution), an analysis of a 5 fold dilution must agree within 10 percent (see 13.2.5.3) of the original determination. Samples identified as Field Blanks cannot be used for serial dilution analysis.

7.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis.

8.0 REQUIRED EQUIPMENT

8.1 Inductively Coupled Plasma-Atomic Emission Spectrometer

8.1.1 Computer controlled atomic emission spectrometer with background correction.

8.1.2 Radio frequency generator.

8.1.3 Argon gas supply, welding grade or better.

8.1.4 Coolflow - or appropriate water cooling device.

9.0 REAGENTS

9.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Re-distilled acids are acceptable. All reagents should be certified by the manufacturer and/or analyzed before use to determine levels of impurities.

9.1.1 Acetic Acid, conc. (sp gr 1.06).

9.1.2 Hydrochloric acid, conc. (sp gr 1.19).

9.1.3 Hydrochloric acid (1+1): Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

9.1.4 Nitric acid, conc. (sp gr 1.41).

9.1.5 Nitric acid (1+1): add 500 mL conc. HNO_3 (sp gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

9.2 Hydrogen Peroxide (30%) (H_2O_2).

9.3 Deionized, distilled water: Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to or better than ASTM Type II reagent water of Specification D 1193.

9.4 Standard stock solutions may be purchased or prepared from ultra high purity grade chemicals or metals. All calibration standards must be traceable to NIST. All salts must be dried for 1 hour at 105° unless otherwise specified.

9.4.1 Recommended sources for stock solutions and mixed solutions are:

9.4.1.1 Inorganic Ventures, Tom's River, NJ (201) 240-6700.

9.4.1.2 Solutions Plus, Inc., Fentum, MO 63026 (314) 349-4922.

9.4.1.3 Spex Industries, Inc., Edison, NJ 08820 (201) 549-7144 or 1-800-LAB-SPEX.

9.4.1.4 National Institute of Standards and Technology (previously NBS), Gaithersburg, MD 20899 (301) 975-4016.

9.4.2 ***CAUTION:*** Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling. Typical stock solution preparation procedures follow:

9.4.2.1 Aluminum solution, stock, 1 mL - 100 μg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

9.4.2.2 Antimony solution, stock, 1 mL - 100 μg Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

9.4.2.3 Arsenic solution, stock, 1 mL - 100 µg AS: Dissolve 0.1320 g of As_2O_3 in 100 mL of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.4 Barium solution, stock, 1 mL - 100 µg Ba: Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 mL deionized, distilled water with 1 ml (1+1) HCl. Add 10.0 mL (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

9.4.2.5 Beryllium solution, stock, 1 mL - 100 µg Be: Do not dry. Dissolve 1.966 g $BeSO_4 \cdot 4H_2O$, in deionized, distilled water, add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.6 Cadmium solution, stock, 1 mL - 100 µg Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.7 Calcium solution, stock, 1 mL - 100 µg Ca: Suspend 0.2498 g $CaCO_3$ (dried at 180°C for 1 hour before weighing) in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.8 Chromium solution, stock, 1 mL - 100 µg Cr: Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete acidify with

10 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.9 Cobalt solution, stock, 1 mL - 100 µg Co: Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3. Add 10.0 ml (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

9.4.2.10 Copper solution, stock, 1 mL - 100 µg Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.11 Iron solution, stock, 1 mL - 100 µg Fe: Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc. HNO_3. Cool, add an additional 5 mL of conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.12 Lead solution, stock, 1 mL - 100 µg Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 mL of conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.13 Magnesium solution, stock 1 mL - 100 µg Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.14 Manganese solution, stock, 1 mL - 100 µg Mn: Dissolve 0.1000 g of manganese metal in the acid mixture, 10 mL conc. HCl and 1 mL conc. HNO_3, and dilute to 1000 mL with deionized, distilled water.

9.4.2.15 Nickel solution, stock, 1 mL - 100 μg Ni: Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1000 mL with deionized, distilled water.

9.4.2.16 Potassium solution, stock, 1 mL - 100 μg K: Dissolve 0.1907 g KCl, dried at 110°C, in deionized, distilled water. Dilute to 1000 mL.

9.4.2.17 Selenium solution, stock, 1 mL - 100 μg Se: Do not dry. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1000 mL.

9.4.2.18 Silver solution, stock, 1 mL - 100 μg Ag: Dissolve 0.1575 g $AgNO_3$ in 100 mL of deionized distilled water and 10 mL conc. HNO_3. Dilute to 1000 mL with deionized, distilled water.

9.4.2.19 Sodium solution, stock, 1 mL - 100 μg Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.20 Thallium solution, stock, 1 mL - 100 μg Tl: Dissolve 0.1303 g $TlNO_3$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.21 Vanadium solution, stock, 1 mL - 100 μg V: Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.4.2.22 Zinc solution, stock, 1 mL - 100 μg Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with deionized, distilled water.

9.5 Mixed calibration standard solutions -- Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see 9.5.1 through 9.5.5). Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl and dilute to 100 mL with deionized, distilled water (see NOTE) in 9.5.5). Prior to preparing the mixed standards, each stock solutions should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change with aging. Calibration standards must be initially verified using a quality control sample and monitored for stability (see 9.7.3). Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 2. Tables 5 and 6 list alternate standard mixes.

9.5.1 Mixed standard solution I -- Manganese, beryllium, cadmium, lead, and zinc.

9.5.2 Mixed standard solution II -- Barium, copper, iron, vanadium, and cobalt.

9.5.3 Mixed standard solution III - Arsenic and selenium.

9.5.4 Mixed standard solution IV -- Calcium, sodium, potassium, aluminum, chromium, and nickel.

9.5.5 Mixed standard solution V -- Antimony, magnesium, silver, and thallium.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized distilled water and warm the flask until the solution clears. Cool and dilute to 100 mL with deionized, distilled water. For this acid combination the sliver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl. Also, depending upon the levels of the elements in the standard mixes, differing acid concentrations may be used.

9.6 Two types of blanks are required for the analysis. The calibration blank (5.12) is used in establishing the analytical curve while the reagent blank (preparation blank, 5.11) is used to correct for possible contamination resulting from varying amounts of acids used in the sample processing.

9.6.1 The calibration blank is prepared in the same concentration of acid as the calibration standards. Prepare a sufficient quantity to be used to flush the system between standards and samples.

9.6.2 The reagent blank (or preparation blank) must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

9.7 In addition to calibration standards, an instrument check standard (5.6), an interference check sample (5.7) and a quality control sample (5.8) are also required for the analyses.

9.7.1 The instrument check standard for continuing calibration verification (CCV) is prepared by the analyst by combining compatible elements at a concentration equivalent to the mid-points of their respective calibration curves. It may be the same source as the calibration standards, or the ICV may be used through the run as the CCV.

9.7.2 The interference check sample (ICS) is prepared by the analyst, or obtained from EPA if available. See Table 8 for required concentrations.

9.7.3 The quality control sample for the initial calibration verification (ICV) should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. EPA will either supply a quality control sample or information where one of equal quality can be procured. It should be a different source than calibration standards.

10.0 CALIBRATION PROCEDURES

10.1 Instruments must be calibrated daily or once every 24 hours and each time the instrument is set up when it is in use. The instrument calibration date and time must be included in the raw data.

10.2 Operating conditions -- Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision,

linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

10.3 Profile and calibrate the instrument according to manufacturer's recommended procedures using mixed calibration standards.

10.4 Analyze the blank and standards 2-4 (suggested standards preparation appears in Tables 5 and 6).

10.4.1 The high standard must be within the proven linear range.

10.4.2 A minimum of two exposures (burns) is required with the average value being used.

10.4.3 Prior to calibration and between each standard and/or sample, flush the system with the calibration blank.

10.5 Once calibration is complete it must be verified by analyzing a calibration verification standard and blank (ICV, ICB) which have been prepared from a different source than the calibration standards. The ICV must be within ±10% of true values to continue to run.

11.0 SAMPLE ANALYSIS

11.1 Perform calibration and verification as described in Section 10.

11.2 Sample analysis is performed according to the run summary listed in Table 7.

11.3 If any samples fall outside the linear range of the instrument, dilute and re-analyze.

12.0 CALCULATIONS

12.1 Reagent (prep) blanks will be analyzed and reported in the appropriate units µg/L (ppb) for water and mg/kg (ppm) for soil; however, no correction of sample results is performed.

12.2 If dilutions are performed, the appropriate factor(s) must be applied to the sample values.

12.3 Prep dilution factors must be applied to the sample values. These include (for solid samples):

12.3.1 Volume (ml)

12.3.2 Weight (g)

12.3.3 Dryness factor (% Solids ÷ 100)

Equation:

$$\text{Sample Conc.} \; \frac{\mu g}{ml} \times \frac{\text{Final Volume (ml)}}{\text{Weight (g)}} \times \frac{1}{\text{Dryness Factor}}$$

$$= \frac{\mu g}{g} = \frac{mg}{kg} \text{ dry weight}$$

12.4 Units must be clearly specified.

12.4.1 Final units for solids are µg/g or mg/kg (ppm) dry weight.

12.4.2 Final units for liquids are µg/L (ppb).

13.0 QUALITY CONTROL

13.1 Quality Control (Run)

Check the instrument calibration by analyzing appropriate quality control check standards as follows:

13.1.1 A quality control sample must be used daily for the initial calibration verification (ICV). This standard must be prepared from a different stock source than the calibration standards. If the results are not within ± 10% of the true value listed for the control sample, prepare a new calibration standards and re-calibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

13.1.2 Analyze the calibration blank (ICB and CCB) at a frequency of 10%. The absolute vale of the result should be less than the contract required detection levels. If the result is not within the control level, terminate the analysis, correct the problem, and re-calibrate the instrument.

13.1.3 For continuing calibration verification (CCV), analyze an appropriate instrument check standard containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ± 10% of the true values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument re-calibrated, and the preceding 10 samples re-analyzed.

13.1.4 To verify inter-element and background correction factors, analyze the ICP interference check samples (9.7.2) at the beginning and end of the sample run or a minimum of twice

per eight-hour work shift, whichever is more frequent. The check samples must be analyzed initially at least five times repetitively to establish a mean value and standard deviation. Results must fall within 80-120% of true values or established mean (exception: silver and antimony). If not, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the samples since the last good ICS.

The levels required for preparation of the interference check Standards are listed in Table 8.

13.1.5 To verify linearity near the CRDL, a standard (CRI) at a level two times the IDL or two times the CRDL, whichever is greater, must be analyzed for every wavelength used for analysis except those for Al, Ba, Ca, Fe, Mg, Na and K. The CRI must be analyzed at the beginning and end of the run or a minimum of twice per 8-hour shift. Specific acceptance criteria have not been set by EPA at the present time.

13.1.6 Quality Control Analysis Scheme (see also Table 7)

13.1.6.1 Calibration.

13.1.6.2 Initial Calibration Verification Standards (ICVA, ICVB, ...).

13.1.6.3 Initial Calibration Blank (ICB).

13.1.6.4 Interference Check Standards (ICSA, ICSAB).

13.1.6.5 2 x CRDL Standard (CRI).

13.1.6.6 Continuing Calibration Verification Standards (CCVA1, CCVB1, ...).

13.1.6.7 Continuing Calibration Blank (CCB1).

13.1.6.8 Sample Prep Blank (PBW (water), PBS (solid)).

13.1.6.9 Laboratory Control Samples (LCSW (water), LCSS (solid)).

13.1.6.10 Analyze eight samples, then run CCVA, CCVB, CCB. Thereafter, run calibration verification standards every tenth sample.

13.1.6.11 For every sample matrix, a 1/5 serial dilution must be run per 20 samples. The serial dilution must be within ± 10% difference of the undiluted sample for diluted sample concentration greater than 50 times the IDL.

13.1.6.12 At the conclusion of the run, the following check samples must be run:

A. CCVA#, CCVB#, ...
B. CCB#
C. ICSA, ICSAB
D. CRI.

13.2 Quality Control (Sample)

13.2.1 Spiked Sample Analysis

13.2.1.1 Pre-digestion spike (matrix spike).

13.2.1.2 At least one spiked sample analysis must be performed on each group of samples of a similar matrix type, for each case of samples, or for each 20 samples received, whichever is more frequent.

13.2.1.3 Samples identified as field blanks cannot be used for spiked sample analysis.

13.2.1.4 Analyte spike levels are specified in Table 9.

13.2.1.5 If spike recovery is not within the limits of 75-125%, all data associated with that spike must be flagged "N" (exception: when sample concentration is greater than 4 times the spike concentration).

13.2.1.6 $$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = spiked sample result
SR = sample results (where SR <IDL, use SR = 0)
SA = spike added

13.2.2 Post Digestion Spikes

13.2.2.1 If the matrix spike recovery does not fall within the 75-125% acceptance limits a post digestion spike must be performed for those elements.

13.2.2.2 Spike the unspiked aliquot of the sample at 2 times the indigenous level or 2 times the CRDL whichever is greater.

13.2.2.3 A post digestion spike is not required for silver.

13.2.2.4 Control limits for post digestion spikes have not at this time, been established by EPA.

13.2.3 Duplicate Sample Analysis

13.2.3.1 At least one duplicate sample must be analyzed from each group of samples of a similar matrix type, for each case of samples, or for each 20 samples received, whichever is more frequent.

13.2.3.2 Samples identified as field blanks cannot be used for duplicate sample analysis.

13.2.3.3 $$RPD = \frac{| D_1 - D_2 |}{(D_1 + D_2)/2} \times 100$$

Where: RPD = relative percent difference
D_1 = first sample value (original sample)
D_2 = second sample value (duplicate)

13.2.3.4 Control limits: ±20% RPD for sample results >5x CRDL
±CRDL for sample results <5x CRDL
±CRDL for one result >5 x CRDL, the other <5x CRDL
if either result <CRDL, RPD is "N.C."

13.2.3.5 Flag all associated results for RPDs which exceed the control limits with an asterisk(*).

13.2.4 Laboratory Control Sample (LCS) Analysis

The LCS must be analyzed for each analyte using the same methods employed for samples (preparation and analysis).

13.2.4.1 Aqueous (LCSW)

13.2.4.1.1 One LCSW must be prepared and analyzed for every 20 samples received, or for each batch of samples digested, whichever is more frequent.

13.2.4.1.2 If results (%R) exceed control limits of 80-120%, analyses must be terminated, the problem corrected, and the samples associated with that LCS re-digested and re-analyzed.

13.2.4.2 Solid LCS (LCSS)

13.2.4.2.1 The availability and use of a LCSS is limited to EPA projects only. An alternate source for the LCSS is being sought.

13.2.4.2.2 Currently, these laboratories are using a liquid concentrate standard reference material with certified values to verify solid sample preparation. It is prepared, using the soil digestion method, with samples at a frequency of one per twenty samples. The certified values are converted to mg/kg using 200 ml/l

g and reported as LCSS. This may change depending on SOW revisions and availability of solid material with control limits.

13.2.4.2.3 If results (%R) exceed control limits of 80-120%, analyses must be terminated, the problem corrected, and the samples associated with that LCS re-digested and re-analyzed.

13.2.5 ICP Serial Dilution Analysis

13.2.5.1 Must be performed on each group of samples of a similar matrix type (i.e., water, soil), for each case of samples, or for each 20 samples received, whichever is more frequent.

13.2.5.2 Samples identified as field blanks cannot be used for serial dilution analysis.

13.2.5.3 An analysis of a 1:5 dilution must agree within 10% difference of the original determination on the undiluted sample when the analyte concentration is minimally a factor of 50x IDL in the original sample.

$$\%D \text{ (percent difference)} = \frac{|I - S|}{I} \times 100$$

Where: I = initial sample result
S = serial dilution result

13.2.5.4 If the original analyte value is not at least 50 times the IDL, that element will not be used in the percent difference determination.

13.2.5.5 If the dilution analysis is not within 10%, the data must be flagged with an "E".

13.3 Quality Control (Quarterly Parameters)

13.3.1 Instrument Detection Limit (IDL) Determination

13.3.1.1 IDLs must be determined prior to the analysis of any field samples and at least quarterly for each instrument.

13.3.1.2 IDLs must meet the Contract Required Detection Limits (CRDL) specified in Table 2.

13.3.1.3 IDLs are three times the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3-5 times the IDL, with seven consecutive measurements per day.

13.3.2 Inter-element Correction Factors

13.3.2.1 Determine as per instrument manufacturer's instructions, and repeat annually.

13.3.3 Linear Range Analysis

13.3.3.1 Linear range verification check standard must be analyzed and reported quarterly for each element.

13.3.3.2 Analytically determined concentration of this standard must be within ±5% of the true value.

13.3.3.3 The concentration of the standard run defines the upper limit of the ICP linear range beyond which results cannot be reported without dilution.

13.3.3.4 When an analyte concentration exceeds the linear range, re-analysis of the prepared sample, after appropriate dilution, is required.

14.0 REPORT FORMAT

14.1 Results will be reported on a standard ITAS Certificate of Analysis unless special requests are made by the client.

Table 1. Inorganic Target Analyte List (TAL)
Contract Required
Detection Limit[1,2]

	(µg/L)	(mg/kg)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	5	1
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.02
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4

[1]If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limits may not equal the Contract Required Detection Limit. This is illustrated in the example below:

For Lead:

Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample Concentration = 220
Contract Required Detection Limit (CRDL) = 5

[2]mg/kg CRDL based on 1.00 g sample brought up to 200 ml.

Table 2. Recommended Wavelengths[2] and Estimated Instrumental Detection Limits

Element	Wavelength, nm[1]	Estimated Detection Limit, µg/l[2]
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Nickel	231.604	15
Potassium	766.491	See [3]
Selenium	196.026	75
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. The use of alternate wavelengths must be reported (in nm) with the sample data.

[2]The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines", EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3]Highly dependent on operating conditions and plasma position.

Table 3. Sample Holding Times, Preservation and Recommended Collection Volumes for Metal Determinations

Measurement	Digestion[a,d] Vol. Req. (ml)	Collection Volume (ml)[b,c]	Preservative	Holding Time
Metals				
Total Recoverable	100	600	HNO_3 to pH <2	6 mo
Dissolved	100	600	Filter on site; HNO_3 to pH <2	6 mo
Suspended	100	600	Filter on site	6 mo
Total	100	600	HNO_3 to pH <2	6 mo

[a]Solid samples are usually digested in 1.00 g aliquots.

[b]Solid samples must be at least 200 g and usually require no preservation other than storing at 4°C until analyzed.

[c]Either plastic or glass containers may be used.

Digestion Procedures:

Water SW-846 Method 3010

Soil/Solid SW-846 Method 3050

[d]Digestion volumes may be altered if insufficient sample is received; however, all reagents used would have to be altered by the same factor.

Table 4. Analytical Concentration Equivalents
Arising From Interference
at the 100 mg/L Level

Analyte	Wavelength (nm)	Interferent[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Cu - 200 mg/L	Mn - 200 mg/L
Ca - 1000 mg/L	Fe - 1000 mg/L	Tl - 200 mg/L
Cr - 200 mg/L	Mg - 1000 mg/L	V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations, to obtain those figures, add the listed concentration to the interferent figure.

Table 5. Calibration Standards Preparation

Standard 1 1% HNO_3 (Standard Blank)

	Element	Stock Concentration (ppm)	Volume (ml)	Standard Concentration (ppm)
Standard 2	Silver	10,000	0.25	10
	Cadmium	10,000	0.25	10
	Cobalt	10,000	0.25	10
	Copper	10,000	0.25	10
	Lead	10,000	0.25	10
	Manganese	10,000	0.25	10
	Nickel	10,000	0.25	10
	Selenium	10,000	0.25	10
	Thallium	10,000	0.25	10
	Vanadium	10,000	0.25	10
Standard 3	Arsenic	10,000	0.25	10
	Barium	10,000	0.25	10
	Beryllium	10,000	0.25	10
	Chromium	10,000	0.25	10
	Antimony	10,000	0.25	10
	Zinc	10,000	0.25	10
Standard 4	Aluminum	10,000	5.0	200
	Calcium	10,000	5.0	200
	Iron	10,000	5.0	200
	Magnesium	10,000	5.0	200
	Potassium	10,000	2.5	100
	Sodium	10,000	2.5	100

Each standard is brought up to 250 ml in 1% HNO_3

Table 6. Check Standards Preparation

ICB/CCB 1% HNO_3 (Calibration Blank)

	Element	Stock Concentration (ppm)	Volume (ml)	Standard Concentration (ppm)
ICVA/CCVA	Aluminum	10,000	1.0	40
	Calcium	10,000	1.0	40
	Iron	10,000	1.0	40
	Potassium	10,000	1.0	40
	Magnesium	10,000	1.0	40
	Sodium	10,000	1.0	40
	Arsenic	1,000	1.0	4
	Barium	1,000	1.0	4
	Beryllium	1,000	1.0	4
	Chromium	1,000	1.0	4
	Antimony	1,000	1.0	4
	Zinc	1,000	1.0	4
ICVB/CCVB	Silver	1,000	1.0	4
	Cadmium	1,000	1.0	4
	Copper	1,000	1.0	4
	Cobalt	1,000	1.0	4
	Manganese	1,000	1.0	4
	Nickel	1,000	1.0	4
	Lead	1,000	1.0	4
	Selenium	1,000	1.0	4
	Thallium	1,000	1.0	4
	Vanadium	1,000	1.0	4

Each standard is brought up to 250 ml in 1% HNO_3

Table 7. Run Summary

I. Calibration

A. Blank (STD1)

B. STD2

C. STD3

D. STD4

II. Analysis

A. ICVA, ICVB (90 to 110% of true value)

B. ICB

C. ICSA, ISCAB (80 to 120% of true value)

D. 2 x CRDL STD (no requirements set to date)

E. CCVA1, CCVB1 (90 to 110% of true value)

F. CCB1

G. Prep Blank

H. LCS (80 to 120% of true value)

I. Run 8 samples (including a 1/5 serial dilution for each matrix)

J. CCVA2, CCVB2

K. CCB2

L. Run 10 samples

M. CCVA#, CCVB#

N. CCB#

O. ICSA, ICSAB

P. 2 x CRDL

Continue L - N until the end of the run, then add O, P.

Table 8. Interferent and Analyte Elemental Concentrations used for ICP Interference Check Sample

Analytes	ICSAB (mg/L)	Interferents	ICSA (mg/L)
Silver	1.0	Aluminum	500
Barium	0.5	Calcium	500
Beryllium	0.4	Iron	200
Cadmium	1.0	Magnesium	500
Cobalt	0.5		
Chromium	0.5		
Copper	0.5		
Manganese	0.5		
Nickel	1.0		
Lead	1.0		
Vanadium	0.5		
Zinc	1.0		

NOTE: If routinely scanning for Arsenic, Selenium, and Thallium it may be useful to include these elements in the ICSAB solution.

Table 9. Spiking Levels for Matrix Spike Sample Analysis

Element	Water	Soil
Aluminum	2,000	*
Antimony	500	500
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Cadmium	50	50
Calcium	*	*
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	*
Lead	500	500
Magnesium	*	*
Manganese	500	500
Nickel	500	500
Potassium	*	*
Selenium	2,000	2,000
Silver	50	50
Sodium	*	*
Thallium	2,000	2,000
Vanadium	500	500
Zinc	500	500

*Not required.

APPENDIX A
EPA METHOD 6010
"TEST METHODS FOR EVALUATING SOLID WASTE PHYSICAL/CHEMICAL METHODS, SW-846, THIRD EDITION, DECEMBER 1987 REVISION"

1.0 PURPOSE

The purpose of this appendix is to outline modifications to the base method in order that the requirements of EPA Method 6010 from "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," SW-846, Third Edition, December 1987 revision can be met.

2.0 MODIFICATIONS TO SCOPE AND APPLICATION

This method extends the analyses to include lithium, molybdenum, phosphorous and strontium. Table 2A has been modified to include these elements with the appropriate analytical wavelength and estimated detection limit.

3.0 MODIFICATIONS TO SUMMARY OF METHOD

No modifications.

4.0 MODIFICATIONS TO SAMPLE HANDLING AND PRESERVATION

Table 3A has been modified to include digestion procedure EPA 3005, otherwise no modifications.

5.0 MODIFICATIONS TO DEFINITIONS

No modifications.

6.0 MODIFICATIONS TO INTERFERENCES

Table 4A has been modified to include molybdenum, otherwise no modifications.

7.0 MODIFICATIONS TO SAFETY

No modifications.

8.0 MODIFICATIONS TO REQUIRED EQUIPMENT

No modifications.

9.0 MODIFICATIONS TO REAGENTS

9.1 Additional standard stock solution recipes follow.

9.1.1 Lithium solution, stock, 1 mL = 100 μg Li: Dissolve 0.5324 g lithium carbonate (mole fraction Li = 0.1878), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HCl and dilute to 1,000 mL with water.

9.1.2 Molybdenum solution, stock, 1 mL = 100 μg Mo: Dissolve 0.20 g $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$ (mole fraction Mo = 0.5772), weighed accurately to at least four significant figures, in water and dilute to 1,000 mL with water.

9.1.3 Strontium solution, stock, 1 mL = 100 μg Sr: Dissolve 0.2415 g of strontium nitrate ($Sr(NO_3)_2$) (mole fraction 0.4140), weighed accurately to at least four significant figures, in a 1-liter flask containing 10 mL of concentrated HCl and 700 mL of water. Dilute to 1,000 mL with water.

9.2 Typical mixed standard solutions are modified to include lithium, molybdenum, phosphorous and strontium.

9.2.1 Mixed standard solution I -- Manganese, beryllium, cadmium, lead, selenium and zinc.

9.2.2 Mixed standard solution II - Barium, copper, iron, vanadium, and cobalt.

9.2.3 Mixed standard solution III -- Arsenic and molybdenum.

9.2.4 Mixed standards solution IV -- Calcium, sodium, potassium, aluminum, chromium, nickel, lithium and strontium.

9.2.5 Mixed standard solution V -- Antimony, magnesium, silver and thallium.

9.3 Preparation of the interference check standard is modified as follows: The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at approximate concentrations of 10 times the instrumental detection limits. In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero. If the particular instrument will display over-correction as a negative number, this spiking procedure will not be necessary.

10.0 MODIFICATIONS TO CALIBRATION PROCEDURES

Calibration verification is modified to include re-analysis of the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5% (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Table 7A is modified to include this analysis.

11.0 MODIFICATIONS TO SAMPLE ANALYSIS

No modifications.

12.0 MODIFICATIONS TO CALCULATIONS

No modifications.

13.0 MODIFICATIONS TO QUALITY CONTROL

13.1 All quality control data should be maintained and available for easy reference or inspection.

13.2 A modified run summary is presented in Table 7A.

13.3 The serial dilution analysis is modified to be a 1/4 dilution for analyte concentration seen at 10 times the instrument detection limit. The 1/4 dilution should agree within ±10% difference of the original sample. If not, a chemical or physical interference effect should be suspected.

13.4 Matrix spiking levels are modified to produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. Table 9 should be modified for each instrument to reflect these ranges. Spike recovery should be 75-125% of the known value. If not, a matrix effect should be suspected.

13.5 Calibration verification is modified to include the re-analysis of the high calibration standard with an agreement of ±5% of established values.

13.6 Calibration blank acceptance is modified to ±3 standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within 3 standard deviations of the background mean, terminate the analysis, recalibrate and re-analyze the previous 10 samples.

13.7 The Original Sample/Duplicate/Spike (OS/D/S) scheme is modified to add a duplicate spike at the same frequency. The new scheme becomes Original Sample/Duplicate/Matrix Spike/Matrix Spike Duplicate (OS/D/MS/MSD).

13.8 The precision control limits are modified to ±20 RPD for sample values greater than 10 times the instrument detection limit.

13.9 The quality control sample should be prepared in the same acid matrix as the calibration standards at 10 times the instrumental detection limits and in accordance with the instructions provided by the supplier.

A quality control reference sample is a sample prepared from an independent standard at a concentration other than that used for calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes of interest from a different source than that used in the preparation of standards for use in the standard curve. A quality control reference sample is intended as an independent check of technique, methodology and standards, and should be run with every analytical batch or every 20 samples, whichever is greater. This is applicable to all inorganic analysis.

NOTE: No acceptance criteria are listed in the method.

13.10 A post-digestion spike is not required.

13.11 A lab control sample is not required. It appears to be replaced with the quality control sample (13.9).

13.12 A CRI standard is not required.

13.13 Method Performance Study

13.13.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table

10A lists the true values, the mean reported values, and the mean percent relative standard deviations.

13.13.2 In a single laboratory evaluation, seven wastes were analyzed for 22 elements by this method. The mean percent relative standard deviation from triplicate analyses for all elements and wastes as 9 ±2%. The mean percent recovery of spiked elements for all wastes was 93 ±6%. Spike levels ranged from 100 µg/L to 100 mg/L. The wastes included sludges and industrial wastewaters.

13.14

~~13.4~~ Instrument detection limit determination as in the base method is not mentioned, however, a method detection limit (MDL) is required and is described below.

For operational purposes, when it is necessary to determine the method detection limit in the sample matrix, the MDL shall be determined by multiplying by 7 the standard deviation obtained from the triplicate analyses of a matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL.

Determine the estimated MDL as follows:

Obtain a concentration value that corresponds to:

a) an instrument signal/noise ratio within the range of 2.5 to 5.0, or

b) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

- Determine the variance (S^2) for each analyte as follows:

$$S^2 = 1/(n-1)\left[\sum_{i=1}^{n} X_i^2 - 1/n \left(\sum_{i=1}^{n} X_i\right)^2\right]$$

- Determine the standard deviation (S) for each analyte as follows:

$$S = (S^2)^{\frac{1}{2}}$$

- Determine the MDL for each analyte as follows:

  $MDL = t_{(n-1,\ 1-\alpha\ =\ 0.99)}\ (S)$

  Where $t_{(n-1,\ 1-\alpha\ =\ 0.99)} = 6.965$ for three replicates as determined from the table of student's t values at the 99 percent level.

14.0 MODIFICATIONS TO REPORT FORMAT

No modifications.

Table 2A. Recommended Wavelengths[2] and Estimated Instrumental Detection Limits

Element	Wavelength, nm[1]	Estimated Detection Limit, µg/l[2]
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Lithium	670.784	5
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Phosphorous	213.618	51
Potassium	766.491	See [3]
Selenium	196.026	75
Silver	328.068	7
Sodium	588.995	29
Strontium	407.771	0.3
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. The use of alternate wavelengths must be reported (in nm) with the sample data.

[2]The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines", EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3]Highly dependent on operating conditions and plasma position.

Table 3A. Sample Holding Times, Preservation and Recommended Collection Volumes for Metal Determinations

Measurement	Digestion[a,d] Vol. Req. (ml)	Collection Volume (ml)[b,c]	Preservative	Holding Time
Metals				
Total Recoverable	100	600	HNO_3 to pH <2	6 mo
Dissolved	100	600	Filter on site; HNO_3 to pH <2	6 mo
Suspended	100	600	Filter on site	6 mo
Total	100	600	HNO_3 to pH <2	6 mo

[a]Solid samples are usually digested in 1.00 g aliquots.

[b]Solid samples must be at least 200 g and usually require no preservation other than storing at 4°C until analyzed.

[c]Either plastic or glass containers may be used.

Digestion Procedures:

Water SW-846 Method 3010 or 3005

Soil/Solid SW-846 Method 3050

[d]Digestion volumes may be altered if insufficient sample is received; however, all reagents used would have to be altered by the same factor.

Table 4A. Analytical Concentration Equivalents Arising From Interference at the 100 mg/L Level

Analyte	Wavelength (nm)	Interferent[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Cu - 200 mg/L	Mn - 200 mg/L
Ca - 1000 mg/L	Fe - 1000 mg/L	Tl - 200 mg/L
Cr - 200 mg/L	Mg - 1000 mg/L	V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations, to obtain those figures, add the listed concentration to the interferent figure.

Table 7A. Run Summary

I. Calibration

A. Blank (STD1)

B. STD2

C. STD3

D. STD4

II. Analysis

A. ICVA, ICVB (90 to 110% of true value) and re-analysis of high calibration standard (95-105% of true value)

B. ICB (establish mean)

C. Interference check standard (80 to 120% of true value)

D. CCVA1, CCVB1 (90 to 110% of true value)

E. CCB1 (±3 std. dev. of the mean blank value)

F. Prep Blank

G. Run 8 samples (including a 1/4 serial dilution for each matrix)

H. CCVA2, CCVB2

I. CCB2

J. Run 10 samples

K. CCVA#, CCVB#

L. CCB#

M. Interference Check Standard

Continue J - L until the end of the run, then add M.

Table 10A. ICP Precision and Accuracy Data[a]

Element	Sample No. 1			Sample No. 2			Sample No. 3		
	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	700	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	50	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se[c]	40	32	21.9	6	8.5	42	10	8.5	8.3

[a]Not all elements were analyzed by all laboratories.

[b]SD = standard deviation.

[c]Results for Se are from two laboratories.

APPENDIX B
EPA METHOD 200.7
"TECHNICAL ADDITIONS TO METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES," EPA-600/4-82-055, DECEMBER 1982.

1.0 PURPOSE

The purpose of this appendix is to outline modifications to the base method in order that the requirements of EPA Method 200.7 from "Technical Additions to Methods for Chemical Analysis of Waster and Wastes," EPA-600/4-82-055, December 1982 can be met.

2.0 MODIFICATIONS TO SCOPE AND APPLICATION

This method extends the analysis to include boron, molybdenum and silica. Table 2B has been modified to include these elements with the appropriate analytical wavelength and estimated detection limit.

3.0 MODIFICATIONS TO SUMMARY OF METHOD

No modifications.

4.0 MODIFICATIONS TO SAMPLE HANDLING AND PRESERVATION

4.1 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pre-treatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter.

4.1.1 For the determination of dissolved elements the sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Use the first 50-100 mL to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

4.1.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

4.1.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) HNO_3 to pH 2 of less as soon as possible, preferable at the time of collection. The sample is not filtered before processing.

5.0 MODIFICATIONS TO DEFINITIONS

No modifications.

6.0 MODIFICATIONS TO INTERFERENCES

Table 4B has been modified to include boron, molybdenum and silicon, otherwise no modifications.

7.0 MODIFICATIONS TO SAFETY

No modifications.

8.0 MODIFICATIONS TO REQUIRED EQUIPMENT

No modifications.

9.0 MODIFICATIONS TO REAGENTS

9.1 Additional standard stock solution recipes follow.

9.1.1 Boron solution, stock, 1 mL = 100 μg B: Do not dry. Dissolve 0.5716 g anhydrous H_3BO_3 in deionized distilled water dilute to 1,000 mL. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

9.1.2 Molybdenum solution, stock, 1 mL = 100 μg Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 mL.

9.1.3 Silica solution, stock, 1 mL = 100 μg SiO_2: Do not dry. Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

9.2 Typical mixed standard solutions are modified to include boron, molybdenum and silicon.

9.2.1 Mixed standard solution I -- Manganese, beryllium, cadmium, lead and zinc.

9.2.2 Mixed standard solution II -- Barium, copper, iron, vanadium and cobalt.

9.2.3 Mixed standard solution III -- Molybdenum, silica, arsenic and selenium.

9.2.4 Mixed standard solution IV -- Calcium, sodium, potassium, aluminum, chromium and nickel.

9.2.5 Mixed standard solution V -- Antimony, boron, magnesium, silver and thallium.

9.3 Preparation of the interference check sample is modified as follows:

The interference check sample is prepared by the analyst in the following manner. Select a representative sample which contains minimal concentrations of the analytes of interest by known concentration of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at the approximate concentration of either 100 μg/L or 5 times the estimated detection limits given in Table 2B. (For effluent samples of expected high concentrations, spike at an appropriate level.) If the type of sample analyzed are varied, a

synthetically prepared sample may be used if the above criteria and intent are met. A limited supply of a synthetic interference check sample will be available from the Quality Assurance Branch of EMSL-Cincinnati.

10.0 MODIFICATIONS TO CALIBRATION PROCEDURES

10.1 Example run summary in modified Table 7B.

10.2 When flushing the system between standards and samples with the calibration blank please note: For boron concentrations greater than 500 μg/L extended flush times of 1 to 2 min. may be required.

10.3 Before beginning the sample run, re-analyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ±5% (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

10.4 If it has been found that methods of standard addition are required, the following procedure is recommended.

10.4.1 The standard addition technique involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_X, are taken. To the first

(labeled A) is added a small volume of V_S of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_S c_s}{(S_A - S_B)V_X}$$

Where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_S is made much less than V_X, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.

2. The chemical form of the analyte added must respond the same as the analyte in the sample.

3. The interference effect must be constant over the working range of concern.

4. The signal must be corrected for any additive interference.

11.0 MODIFICATIONS TO SAMPLE ANALYSIS

No modifications.

12.0 MODIFICATIONS TO CALCULATIONS

12.1 Reagent blanks should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

12.2 Data should be rounded to the thousandth place and all results should be reported in mg/L up to three significant figures.

13.0 MODIFICATIONS TO QUALITY CONTROL

13.1 The calibration verification must agree within ±5% of true value.

13.2 The calibration blank must be within 2 standard deviations of the mean value.

13.3 The interference check sample must be within 1.5 standard deviations of the mean value.

13.4 A quality control sample obtained from an outside source must be used as the ICV. A fresh dilution of this sample shall be analyzed weekly thereafter to monitor their stability. If the results are not within ±5% of the true value listed for the control sample, prepare a new calibration standard and re-calibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

13.5 Matrix Spike

The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect. *Caution:* The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

13.6 A lab control sample is not required.

13.7 A CRI standard is not required

13.8 Instrumental Detection Limit

The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

13.9 Method Precision and Accuracy Study

In an EPA round-robin phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table 10B lists the true value, the mean reported value and the mean % relative standard deviation.

14.0 MODIFICATIONS TO REPORT FORMAT

No modifications.

Table 2B. Recommended Wavelengths[2] and Estimated Instrumental Detection Limits

Element	Wavelength, nm[1]	Estimated Detection Limit, µg/l[2]
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	See [3]
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. The use of alternate wavelengths must be reported (in nm) with the sample data.

[2]The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines", EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3]Highly dependent on operating conditions and plasma position.

Table 4B. Analytical Concentration Equivalents Arising From Interference at the 100 mg/L Level

Analyte	Wavelength (nm)	Interferent[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Boron	249.773	0.04	-	-	-	0.32	-	-	-	-	-
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Silicon	288.158	-	-	0.07	-	-	-	-	-	-	0.01
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Cu - 200 mg/L	Mn - 200 mg/L
Ca - 1000 mg/L	Fe - 1000 mg/L	Tl - 200 mg/L
Cr - 200 mg/L	Mg - 1000 mg/L	V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations, to obtain those figures, add the listed concentration to the interferent figure.

Table 7B. Run Summary

I. Calibration

A. Blank (STD1)

B. STD2

C. STD3

D. STD4

II. Analysis

A. ICVA, ICVB (95 to 105% of true value) and re-analyze the highest calibration standard (95-105% of true value)

B. ICB

C. Interference check standard (±1.5 standard deviations of the mean)

D. CCVA1, CCVB1 (95 to 105% of true value)

E. CCB1 (±2 standard deviations of the mean)

F. Prep Blank

G. Run 8 samples (including a 1/5 serial dilution for each matrix)

H. CCVA2, CCVB2

I. CCB2

J. Run 10 samples

K. CCVA#, CCVB#

L. CCB#

M. Interference Check Standard

Continue J - L until the end of the run, then add M.

Table 10B. ICP Precision and Accuracy Data[a]

Element	Sample No. 1			Sample No. 2			Sample No. 3		
	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)	True Value (µg/L)	Mean Reported Value (µg/L)	Mean SD[b] (%)
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	500	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se[c]	40	32	21.9	6	8.5	42	10	8.5	8.3

[a] Not all elements were analyzed by all laboratories.

[b] SD = standard deviation.

[c] Results for Se are from two laboratories.

ITAS CINCINNATI LABORATORY

PROCEDURE/DOCUMENT CHANGE

PROCEDURE/DOCUMENT TITLE AND NUMBER: Analysis of Volatile Organic Compounds by GC/MS for CLP

SOP No.: 10-001-00

PROCEDURE/DOCUMENT SECTION(S) AFFECTED BY CHANGE:

5.3.7.1, 5.3.7.2, 5.3.7.4, 5.3.9.1, 6.2.2

REASON FOR CHANGE OR ADDITION: The use of 25 ml purge volume for water samples to achieve lower detection limits. Use of bromoform and 1,1,2,2-Tetrachloroethane as SPCC's not feasible with this approach.

CHANGE EFFECTIVE FROM: 8/6/90 (DATE) TO: Project Completion (DATE)

SAMPLES OR PROJECTS AFFECTED: Wright Patterson Air Force Base (Battelle ERIO Groundwater Investigation)

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY):

5.3.7.1 - concentration levels to be used are 4, 10, 20, 30, 40 µg/L

5.3.7.2 - addition of 5µl of this STD to 25ml of sample or calibration STD is equivelent to 10µg/L 5.3.7.4 - SPCC compounds are chloromethane, 1,1-dichloroethane & Chlorobenzen

5.3.9.1 - (5) adjust the sample vol. to 25.0 ml

6.2.2 - calibration is required at 4, 10, 20, 30, 40

SUBMITTED BY: [signature] DATE: 8/6/90

, TECHNICAL SPECIALIST

[signature] 8-6-90, LABORATORY MANAGER

Lauren Driess 8-7-90, QC COORDINATOR

Title:

Analysis of Volatile Organic Compounds by GC/MS for CLP

Word Processing Disk Number: 1850

Prepared by	Approved by	Date	QA Concurrence	Date
		8-7-89		9/7/89

1.0 PURPOSE

To describe the procedure for the analysis of water, sediment and soil samples for the organic compounds on the Target Compounds List (TCL) (see Table 1).

2.0 SCOPE/APPLICATION

This method is divided into the following sections: sample preparation, screening, and analysis. Sample preparation covers sample storage, sample holding times, and medium level sample extraction. The screening section contains a technique for estimation of levels of organic materials present in samples. The analysis section contains the GC/MS analytical methods for organics. The purge and trap technique, including related sample preparation, is included in the analysis section.

All CLP VOA analyses are done using this method. Forms to be used in reporting analytical and QC data are attached.

3.0 References

3.1 EPA Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, 1982, Method 624.

3.2 EPA SW-846, 3rd ed., Method 8240.

3.3 CLP Statement of Work Rev. 2/88.

3.4 PEI Laboratory Quality Assurance Manual.

Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)*

Volatiles	CAS number	Quantitation limits** Water, µg/L	Low soil/sediment,[a] µg/kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-34-3	5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,2-Dichloropropane	78-87-5	5	5
19. cis-1,3-Dichloropropene	10061-01-5	5	5
20. Trichloroethene	79-01-6	5	5
21. Dibromochloromethane	124-48-1	5	5
22. 1,1,2-Trichloroethane	79-00-5	5	5
23. Benzene	71-43-2	5	5
24. trans-1,3-Dichloropropene	10061-02-6	5	5
25. Bromoform	75-25-2	5	5
26. 4-Methyl-2-pentanone	108-10-1	10	10
27. 2-Hexanone	591-78-6	10	10
28. Tetrachloroethene	127-18-4	5	5
29. Toluene	108-88-3	5	5
30. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
31. Chlorobenzene	108-90-7	5	5
32. Ethyl Benzene	100-41-4	5	5
33. Styrene	100-42-5	5	5
34. Xylenes (Total)	1330-20-8	5	5

[a] Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Volatile TCL Compounds are 125 times the individual Low Soil/Sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitatin limits calcualted by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract will be higher.

Table 1

4.0 Associated SOPs

None

5.0 Procedure

5.1 Sample Storage and Holding Times

5.1.1 Procedures for Sample Storage - The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until analysis or extraction.

5.1.2. Contract Required Holding Times - VOA analysis of water or soil/sediment samples must be completed within 10 days of receipt for CLP analyses.[1]

5.2 Sample Screening - Samples may be screened for organic content with the Photovac TIP 1 portable photoionizaiton detector.

5.2.1 Summary of Method - The soil/sediment and water samples may be screened for hydrocarbon content with the Photovac TIP 1 portable air analyzer. The results of the screening will determine if samples are to be analyzed by low or medium level GC/MS procedures for soil samples or to determine the appropriate dilution factor for water samples.

5.2.2 Apparatus - Detector. Photovac TIP 1 portable photoionization detector. The detector has an illuminated LCD display which shows the response, as well as a zero control knob and a sensitivity adjust knob. The sensitivity knob allows the analyst to adjust the sensitivity of the detector depending on the concentration of the samples. The sensitivity increases with increase in the setting; thus a setting of zero results in no sensitivity for very concentrated samples and a setting of 9 results in the highest sensitivity, for very dilute samples. It should be noted that adjusting the sensitivity will alter the response of the calibration standards.

[1]When water samples are unpreserved (i.e., no HCl), VOA analyses must be completed within 5 days.

5.2.3 Sample Screening - The analyzer is calibrated using standards of various levels of toluene in water. The response for each level is noted and a calibration curve is calculated.

5.2.3.1 Preparation of Standards

Toluene standards are prepared by adding toluene to deionized water. Concentrations range from 0 ppb to 100,000 ppb. The table below details the preparation of these standards.

Toluene	Amount used	Deionized water	Final conc.
Neat	11.5 ul	100 ml	100,000 ppb
100,000 ppb std.	1 ml	10 ml	10,000 ppb
100,000 ppb std.	100 ul	10 ml	1,000 ppb
100,000 ppb std.	50 ul	10 ml	500 ppb
100,000 ppb std.	20 ul	10 ml	200 ppb
100,000 ppb std.	10 ul	10 ml	100 ppb

A blank is prepared from pure deionized water. Each standard is prepared in volumetric flasks and then transferred to small vials (1.5 ml-3.5 ml) with no headspace. They are then stored in a refrigerator designed for volatile organics until ready to be analyzed.

5.2.3.2 Calibration. Approximately 1.5 ml of each standard is added to a 3.5 ml vial. The vials are shaken and set aside for a few minutes to allow the headspace to equilibrate. Air is drawn from the headspace above the standard and the highest response noted. (Care must be taken so that none of the liquid is drawn into the analyzer.) Be sure to also analyze a blank water.

Construct a calibration curve plotting response vs. concentration for the standards.

5.2.3.3 Estimation of Concentration of Volatile Organics in Samples. Approximately 1.5 g of the sample, soil or water, is placed in a 3.5 ml vial. The vial is then capped and allowed to equilibrate for 15-20 minutes. The cap is then removed and the analyzer probe tip is inserted into the mouth of the vial. The highest analyzer response is noted and compared to the calibration curve. An approximate starting dilution can then be calculated. (Use a dilution calculated to have a final concentration of approximately 100 ppb.) Note: The TIP analyzer is 10-100 times more sensitive to aromatic materials than to chlorinated hydrocarbons or ketones.

5.3 GC/MS Analysis of Volatile Organic Compounds (VOA)

5.3.1 Water Samples Method Summary

An inert gas is bubbled through a 5 mL sample contained in a specially designed purging chamber (Figure 1) at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column (Figure 2) where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging. All water samples are, by definition, low level.

Figure 1

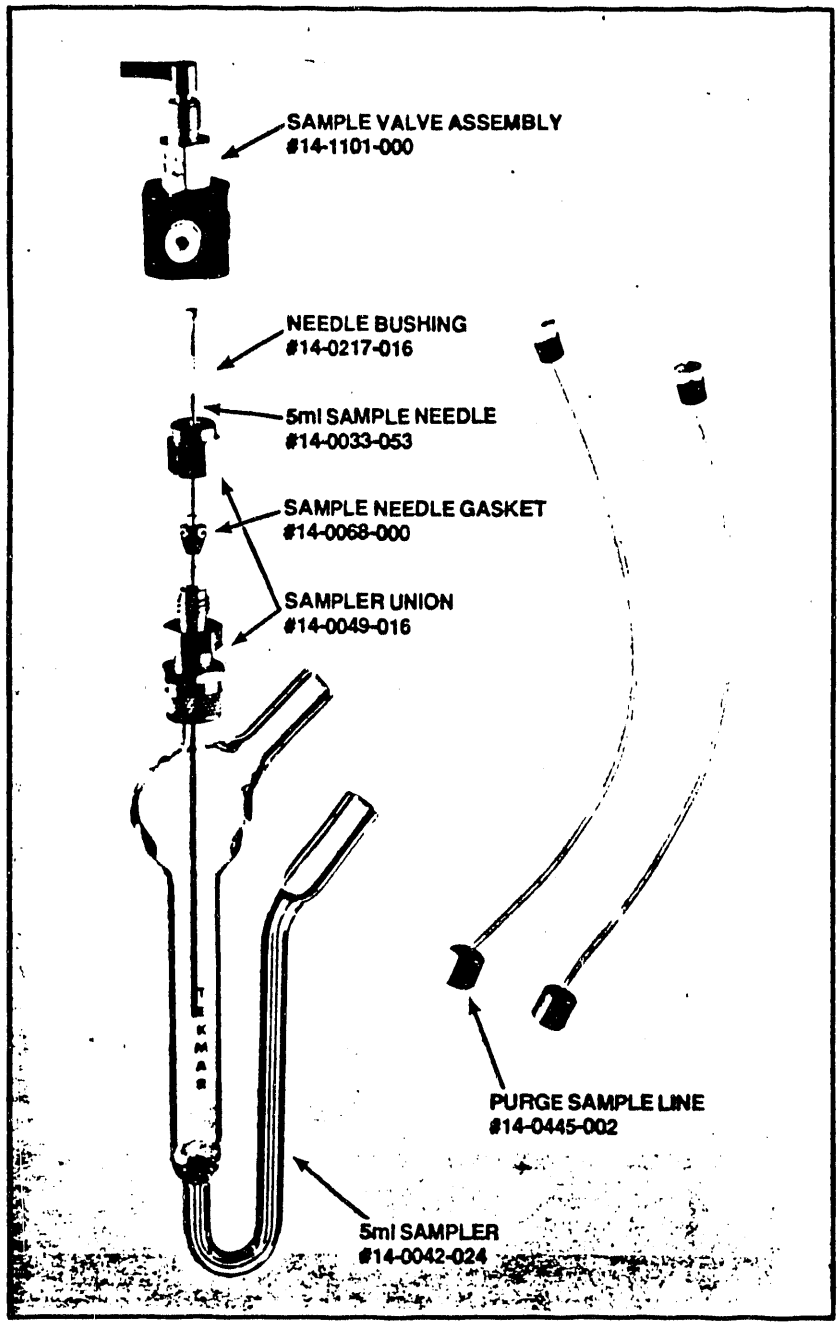
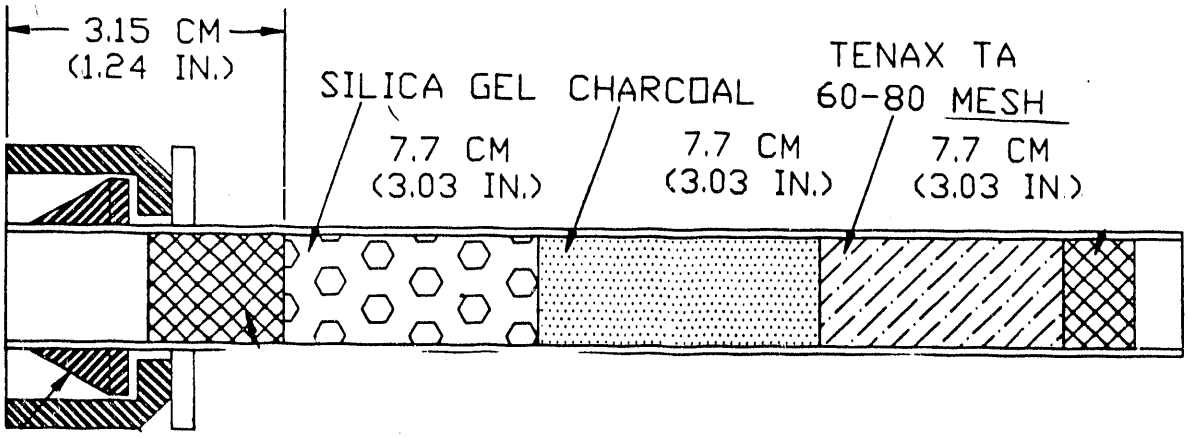
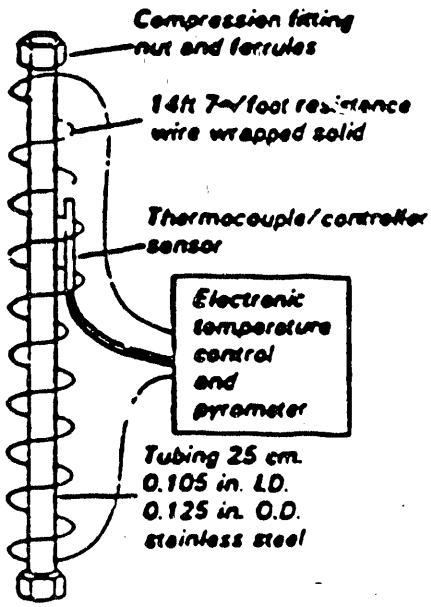

SAMPLE VALVE ASSEMBLY
#14-1101-000
NEEDLE BUSHING
#14-0217-016
5ml SAMPLE NEEDLE
#14-0033-053
SAMPLE NEEDLE GASKET
#14-0068-000
SAMPLER UNION
#14-0049-016
PURGE SAMPLE LINE
#14-0445-002
5ml SAMPLER
#14-0042-024

Figure 2


3.15 CM
(1.24 IN.)
SILICA GEL
7.7 CM
(3.03 IN.)
CHARCOAL
7.7 CM
(3.03 IN.)
TENAX TA
60-80 MESH
7.7 CM
(3.03 IN.)


Compression fitting nut and ferrules
14ft 7-√foot resistance wire wrapped solid
Thermocouple/controller sensor
Electronic temperature control and pyrometer
Tubing 25 cm. 0.105 in. I.D. 0.125 in. O.D. stainless steel

5.3.2 Soil/Sediment Samples Method Summary

5.3.2.1 Low Level. An inert gas is bubbled through a mixture of a 5 g sample (weighed to the nearest 0.1 g) and 15 ml reagent water contained in a suggested specially designed purging chamber (Figure 2A) at 40°C. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

5.3.2.2 Medium Level. A measured amount of soil (4 g ± 0.1 g) is extracted with 10 ml of methanol. An aliquot of the methanol extract is added to 5 mL of reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber (Figure 1) at ambient temperature. The purgeables are effectively transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

5.3.3 Interferences

Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in

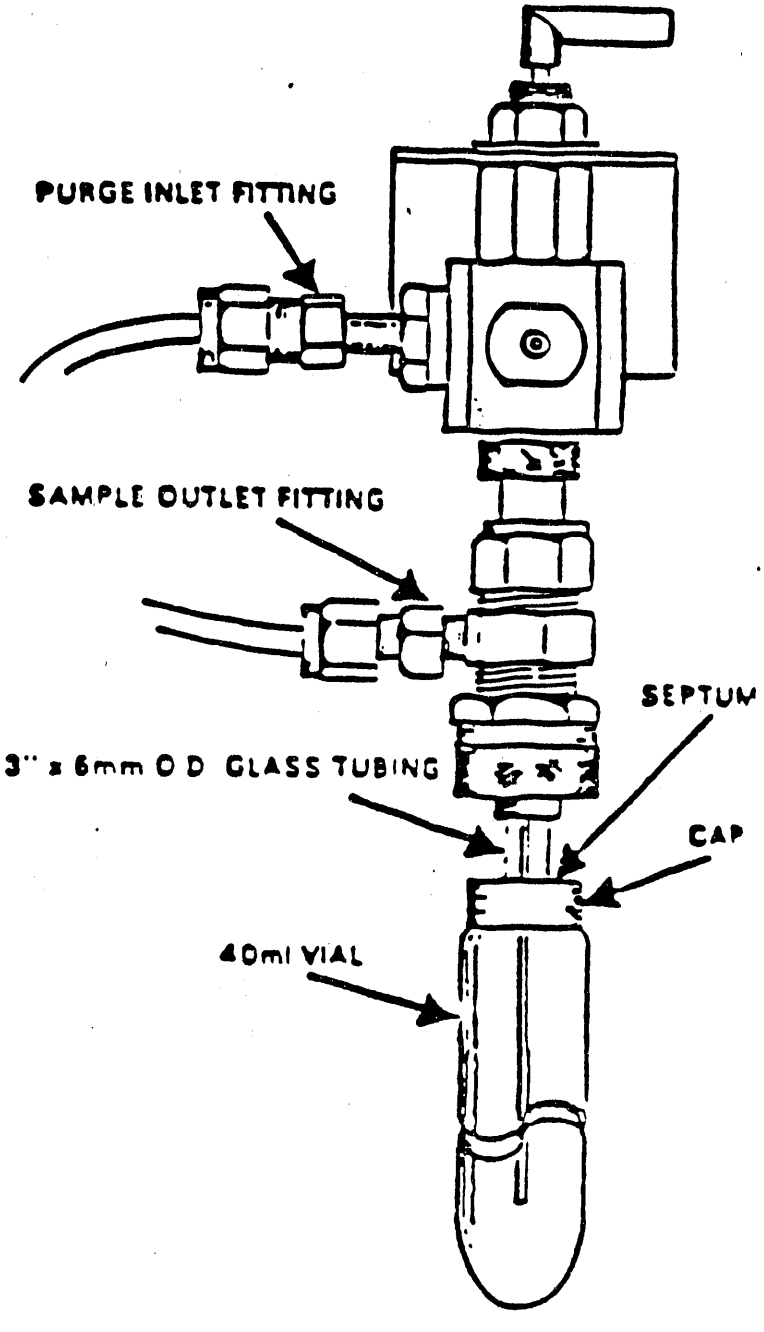


Figure 2A. Low soil impinger.

the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. If methanol dilutions are to be analyzed, a methanol blank must also be analyzed. No samples can be analyzed until the system is demonstrated to be free of contamination.

Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check on such contamination. One holding blank per case should be analyzed. Data must be retained in the laboratory and kept available for inspection.

Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

The laboratory where volatile analysis is performed should be kept as free of solvents as possible.

5.3.4 Apparatus and Materials

5.3.4.1 Micro syringes 10 uL and larger, 0.006 inch ID needle.

5.3.4.2 Syringe valve - two-way, with Luer ends (three each), if applicable to the purging device.

5.3.4.3 Syringe - 5 mL, gas tight with shut-off valve.

5.3.4.4 Balance-Analytical, capable of accurately weighing ±0.0001 g. and a top-loading balance capable of weighing ±0.1 g.

5.3.4.5 Glassware

- Water purge vessel.
- Soil purge vessel.
- Bottle - 15 mL, screw cap, with Teflon cap liner.
- Volumetric flasks - class A with ground-glass stoppers.
- Vials - 3.5 mL for TIP screening.

5.3.4.6 Purge and trap - The purge and trap device consists of a Tekmar model ALS autosampler or model 4210 interfaced with a Tekmar model LSC-2 thermal desorber.

The sample purger for water samples is designed to accept 5 mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less then 3 mm at the origin. The sample purger, illustrated in Figure 1, meets these design criteria. These purge vessels are available from Tekmar. Alternate sample purge devices may be utilized provided equivalent performance is demonstrated. An example of a suitable

Figure 3

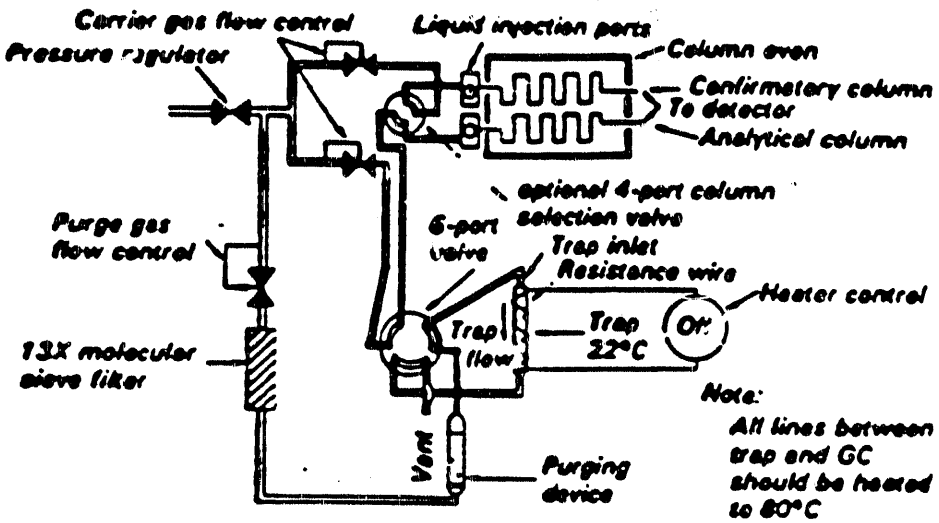


Figure 3. *Schematic of purge and trap device — purge mode*

impinger for low level soil samples is illustrated in Figure 2a.

The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following lengths of absorbents: 7.7 cm of 2,6-diphenylene oxide polymer (Tenax-GC 60/80 mesh), 7.7 cm of charcoal, and 7.7 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent). The minimum specifications for the trap are illustrated in Figure 2.

The desorber should be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 220°C during bakeout mode. The desorber design, illustrated in Figure 3, meets these criteria.

The transfer line from the purge and trap unit is coupled directly to the injection port of the gas chromatograph.

For low level soil samples, the purge vessel is immersed in a heated bath capable of maintaining the samples at 40°C ±1°C.

5.3.4.7 GC/MS

Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

The temperature program used is: initial temperature 45°C, hold for 3 minutes, then ramp to 170°C at 6°C/min. The injection port temperature is 210°C. Carrier gas is helium (60 psi).

Column - 30 meter long x 0.53 mm ID Fused Silica Capillary Column coated with a 2 micron film thickness of DB-624 or equivalent. Alternately, a 6 ft. x 2 mm ID glass column packed with 1.0% SP-1000 on Carbopack B (60/80 mesh) with a 3 inch precolumn of 3% SP-1000 on 100/120 Supelcoport may be used.

Mass spectrometer - Scanning from 40 to 260 amu every 3 seconds (or faster), utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the following criteria in Table 2 when 50 ng of p-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

TABLE 2. BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0 percent of mass 174
174	Greater than 50.0 percent of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

GC/MS interface - A glass jet separator is used to interface the gas chromatograph and mass spectrometer.

Data system - A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for

ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available to integrate the abundance in any EICP between specified time or scan number limits.

5.3.5 Reagents

5.3.5.1 Reagent water. Reagent water is prepared by boiling water from the millipore water purification system. This water is maintained at a low boil until needed. It is then poured into glass vessels which are kept in an oven at 50°C prior to use needed. The vessel is quickly cooled by immersion in an ice water bath.

Methanol. Pesticide quality or equivalent, B&J Purge and Trap Methanol.

5.3.6 Standards

Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at 4°C (±3°C) in screw-cap amber bottles with Teflon liners.

5.3.6.1 Stock standard solutions. Stock standard solutions are purchased from Supelco and are compared to EPA respiratory standards before use.

Prepare fresh working standards every two weeks for gases or for reactive compounds such as styrene. All other standards must be replaced after six months, or sooner if comparison with check standards indicates a problem.

5.3.6.2 Surrogate standard spiking solution. Stock standard solutions for toluene-d8, p-bromofluorobenzene, and 1,2-dichloroethane-d4 in methanol are also purchased from Supelco and diluted to 50 ng/ul for use.

5.3.6.3 Internal standard spiking solution. 50 ug/ml of each bromochloromethane, 1,4-difluorobenzene and chlorobenzene-d_5.

5.3.6.4 Purgeable Organic Matrix Standard Spiking Solution. Prepare a spiking solution in methanol that contains the following compounds at a concentration of 50 ng/ul;

Purgeable organics

1,1-dichloroethene
Trichloroethene
Chlorobenzene
Toluene
Benzene

Matrix spikes also serve as duplicates; therefore, add an aliquot of this solution to each of two portions from one sample chosen for spiking.

5.3.6.5 BFB Standard. Prepare a 50 ng/ul solution of BFB in methanol.

5.3.7 Calibration

Assemble the purge and trap device and connect it to the injection port of the gas chromatograph. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 cm^3/min.

Capillary Column - Gas Chromatographic Conditions:

Temperature program: -30°C initial temperature. Zero hold time. Program at 12°C/min. to 150°C. Hold for 5 minutes.
Injection port: 210°C
Purge flow: 20-30 cc/min.
Desorb (carrier) flow: 8 cc/min.
Interface oven: 240-270°C.

Gas chromatographic conditions are as follows for packed columns:

Temperature program: 45° initial temperature. Hold for 3 min. then program to 240°C at 6°/min.
Injection port temperature: 210°C
Purge flow: 20-30 cc/min.
Desorb (carrier) flow: 20 cc/min.
Interface oven temperature: 240-270°C

Internal standard calibration procedure. The three internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5, at 50 ug/L at time of purge. Separate initial and continuing calibrations must be performed for water samples, low level soil samples, and medium level soil samples.

5.3.7.1 Prepare calibration standards at five concentration levels for each TCL parameter and each surrogate compound. The concentration levels to be used are 20, 50, 100, 150 and 200 ug/l.

5.3.7.2 Prepare a spiking solution containing the internal standards using the procedures described in Section 5.3.6. A secondary dilution standard is prepared at a concentration of 50 ug/ml of each internal standard compound. The addition of 5 uL of this standard to 5 ml of sample or calibration standard is equivalent of 50 ug/L.

5.3.7.3 Tune the GC/MS system to meet the criteria on Page 14 by injecting BFB. Analyze each calibration standard, adding 5 ul of internal standard spiking solution directly to the syringe. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate relative response factors (RRF) for each compound using equation 1.

Eq. 1 $$RRF = \frac{A_x}{A_{is}} \quad \frac{C_{is}}{C_x}$$

Where:

Ax = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard from pages 33 and 34.

C_{is} = Concentration of the internal standard.

C_x = Concentration of the compound to be measured.

5.3.7.4 The average relative response factor (RRF) must be calculated for all compounds, including surrogates. A system performance check must be made before this calibration curve is used. Five compounds (the system performance check compounds) are checked for a minimum average relative response factor. These compounds (the SPCC) are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. Six compounds (the calibration check compounds, CCC) are used to evaluate the curve. These compounds the (CCC) are 1,1-Dichloroethane, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve. A minimum %RSD for each CCC must be met before the curve is valid.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

All data are entered on the Initial Calibration Data Form (Form VI).

5.3.7.5 Check of the calibration curve must be performed once every 12 hours. These criteria are described in detail in Form VII, Continuing Calibration Check. The minimum relative response factor for the system performance check compounds must be checked. If this criteria is met, the relative response factor of all compounds are calculated and reported. A percent difference of the daily relative response factor (12 hour) compared to the average relative response factor from the initial

curve is calculated. The maximum percent difference allowed for each compound flagged as 'CCC' in Form VII is checked. Only after both these criteria are met can sample analysis begin.

5.3.7.6 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is necessary. Both internal standard areas and retention times are plotted on control charts immediately after data processing.

5.3.8 GC/MS Operating Conditions

These performance tests require the following instrumental parameters:

Electron energy:	70 Volts (nominal)
Mass range:	40-260
Scan time:	To give at least 5 scans per peak and not to exceed 3 seconds per scan

5.3.9 Sample Analysis

5.3.9.1 Water Samples

1) All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

2) Operating conditions for the gas chromatograph are listed in Section 5.3.7.

3) After achieving the key ion abundance criteria using BFB, calibrate the system with either the initial calibration procedure or continuing calibration procedure.

4) Adjust the purge gas (helium) flow rate to 25-40 cm^3/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

5) Remove a 5 mL syringe barrel from the oven and attach a closed syringe valve. Open the sample, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the sample for future analysis so if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as when the analyst has determined that the first sample has been analyzed properly. Filling one 20 mL syringe would allow the use of only one syringe. If a second analysis is needed from the 20 mL syringe, it must be analyzed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

6) The purgeable organics screening procedure (Section 5.2), will have shown the approximate concentrations of major sample components. If a dilution of the sample was indicated, this dilution shall be made just prior to GC/MS analysis of the sample. All steps in the dilution procedure must be performed without delays until the point at which the diluted sample is in a gas tight syringe.

The following procedure will allow for dilutions near the calculated dilution factor from the screening procedure:

All dilutions are made in volumetric flasks (10 mL to 100 mL).

Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

Inject the proper aliquot from the syringe into the volumetric flask. Aliquots of less than 1 mL increments are prohibited. Dilute the flask to the mark with reagent water. Cap the flask, invert, and shake three times.

Fill a 5 mL syringe with the diluted sample.

If this is an intermediate dilution, use it and repeat above procedure to achieve larger dilutions.

7) Add 5 ul of the surrogate spiking solution (page 13) and 5 ul of the internal standard spiking solution (page 13) through the valve bore of the syringe, then close the valve.

Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

8) Close both valves and purge the sample for 11.0 ± 0.1 minutes at ambient temperature.

9) At the conclusion of the purge time, switch the device to the desorb mode, and begin the gas chromatographic temperature program. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with helium at 20 ml/min for four minutes.

10) While the trap is being desorbed into the gas chromatograph, empty the purging chamber if operating in manual mode. Wash the chamber with a minimum of two 5 mL flushes of reagent water to avoid carryover of pollutant compounds.

11) After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C. Trap temperatures up to 220°C may be employed, however the higher temperature will shorten the useful life of

the trap. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

12) If the initial analysis of a sample or a dilution of a sample has a concentration of TCL compounds that exceeds the initial calibration range, the sample must be reanalyzed at a greater dilution.

13) For water samples, add 10 uL of the matrix spike solution (page 13) to the 5 mL of sample purged. Disregarding any dilutions, this is equivalent to a concentration of 40 ug/L of each matrix spike compound.

14) All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the calibration curve.

5.3.9.2 Soil/Sediment Samples

Screening of samples is recommended to determine if dilutions will be required. If peaks are saturated from the analysis of a 5 g sample, a smaller sample size must be analyzed to prevent saturation. However, the smallest sample size permitted is 1 g. If smaller than 1 g sample size is needed to prevent saturation, the medium level method must be used.

1) Low Level Soil Method The low level soil method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards under the same conditions as the

samples. An especially designed purge vessel is used for these analyses (see Figure 2A). Screen the sample before analysis.

The GC/MS system should be set up as previously discussed. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and sample. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions given except for the addition of a 40°C purge temperature.

To prepare the reagent water containing the surrogates and internal standards, remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 5 ul each of the surrogate spiking solution and the internal standard solution to the syringe through the valve. The addition of 5 uL of the surrogate spiking solution to 5 g of soil/sediment is equivalent to 50 ug/kg of each surrogate standard.

The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined from screening into a

tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

Immediately after weighing the sample, if the sample is for VOA anlaysis only, determine pH of sample with pH indicating paper prior to taring for percent moisture determination. Weigh 5 to 10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\text{Percent moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

If the sample is also being analyzed for BNA's or pests, % moisture and pH will be determined by the prep lab.

Add 10 ml of reagent water to the purge vessel, cap, shake to disperse the sample, and attach it to the ALS unit. Spike a syringe containing 5 ml of reagent water with internal standards and surrogates, and inject through the valve into the sample.

Immerse the purge vessel in a 40°C ±1°C water bath, and purge the sample for 11.0 ±0.1 minutes.

Proceed with the analysis as for water samples. Use 10 mL of the same reagent water for the reagent blank.

For low level soils/sediment add 5 ul of the matrix spike solution to the 10 mL of water. The concentration for a 5 g sample would be equivalent to 50 ug/kg of each matrix spike standard.

2) Medium Level Soil Method. The medium level soil method is based on extracting the soil/sediment sample with methanol. An aliquot of the methanol extract is added to reagent water containing the surrogate and internal standards and purged at ambient temperature. All samples with a dilution factor greater than 5.0 should be analyzed by the medium level method. If saturated peaks or concentrations exceeding the calibration range occurred or would occur when a 1 g sample was analyzed, the medium level method must be used.

The GC/MS system should be set up as previously discussed. This should be done prior to the addition of the methanol extract to reagent water. Initial and continuing calibrations are performed by adding standards in methanol to reagent water and purging at ambient temperature.

The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) into a tared 15 mL vial. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g. Also

determine the percent moisture and pH if the sample is for VOA analysis only (see Section 5.3.9.2).

Quickly add 9.0 mL of methanol, then 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes. NOTE: Perform rapidly to avoid loss of volatile organics. Perform in a laboratory free of solvent fumes.

Using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be disposed of. Transfer approximately 1 mL of the reagent methanol to a GC vial for use as the method blank for each case or set of 20 samples, whichever is more frequent. These extracts may be stored in the dark at 4°C (±2°C) prior to analysis. Use the dilution factor determined by the screening to obtain the proper aliquot for analysis.

The volume of methanol added to the 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

Remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5 mL to

allow volume for the addition of sample and standards. Add 5 uL of the internal standard solution.

Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber. A water purge vessel is used for analysis of methanol dilutions (Figure 1).

Proceed with the analysis. Analyze all reagent blanks on the same instrument as the samples. The standards should also contain 100 uL of methanol to simulate the sample conditions.

For a matrix spike in the medium level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution, and 1.0 mL of matrix spike solution. This results in a 6,200 ug/kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100 uL aliquot of this extract to 5 mL of water for purging.

5.3.10 Qualitative Analysis

The compounds listed in the Target Compound List (TCL) in Table 1 shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: (1) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

5.3.10.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

5.3.10.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the same GC/MS may be used for identification purposes, only if the GC/MS meets the daily tuning requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs.

5.3.10.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of ions must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should

favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by a "J," e.g., "3J."

5.3.10.4 If a compound cannot be verified by all of the criteria above, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification.

5.3.10.5 A library search shall be executed for non-TCL sample components for the purpose of tentative identification for CLP analyses or where requested. For this purpose, the 1985 release of the National Bureau of Standards Mass Spectral Library (or more recent release), containing 42,261 spectra, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Up to 10 nonsurrogate, non target organic compounds of greatest apparent concentration shall be tentatively identified via a forward search of the NBS mass spectral library. (Substances with responses less than 10% of the internal standard are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library

search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Guidelines for making tentative identification:

Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

If in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid type,

unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

5.3.11 Quantitative Analysis

5.3.11.1 TCL components identified shall be quantified by the internal standard method. The internal standard used shall be that which is listed in Table 2. The EICP area of the characteristic ions of analytes listed in Tables 2 and 3 in this section are used.

TABLE 2. CHARACTERISTIC IONS FOR SURROGATE AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

Parameter	Primary ion	Secondary ion(s)
Surrogate standards		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane d-4	65	102
Toluene d-8	98	70, 100
Internal standards		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene d-5	117	82, 119

TABLE 3. CHARACTERISTIC IONS FOR VOLATILE TCL COMPOUNDS

Parameter	Primary ion*	Secondary ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethane	96	61, 98
1,1-Dichloroethene	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	72	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Vinyl acetate	43	86
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	-
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100

(continued)

TABLE 3 (continued)

Parameter	Primary ion*	Secondary ion(s)
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

5.3.11.2 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

If after re-analysis, the EICP areas for all internal standards are inside the required limits (-50% to +100% of that internal standard in the

mid low standard analysis for that day), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only submit data from the analysis with EICP's within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

If the re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables. Document in the Case Narrative all inspection and corrective actions taken.

5.3.11.3 The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. Use the relative response factor and the equations below. When TCL compounds are below contract required quantitation limits (CRQL) but the spectra meet the identification criteria, report the concentration with a "J." For example, if CRQL is 10 ug/L and concentration of 3 ug/L is calculated, report as "3J."

5.3.11.4 Xylenes (o,m, & p - isomers) are to be reported as xylenes (total). Since o- and p-xylene coelute, the xylenes must be quantitated as m-xylene. The concentration of all xylene isomers must be added together to give the total.

1,2-Dichloroethene (trans and cis stereoisomers) are to be reported as 1,2--Dichloroethene (total). The concentrations of both isomers must be added together to give the total.

5.3.12 Calculations

5.3.12.1 Water

$$\text{Concentration} \quad \text{ug/L} = \frac{(A_x)(I_s)}{(A_{is})(RRF)(V_o)}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard from Exhibit E.
I_s = Amount of internal standard added in nanograms (ng).
V_o = Volume of water purged in milliliters (mL) (take into account any dilutions).

5.3.12.2 Sediment/Soil (medium level)

$$\text{Concentration (Dry weight basis)} \quad \text{ug/k} = \frac{(A_x)\ (I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

5.3.12.3 Sediment/Soil (low level)

$$\text{Concentration (Dry weight basis)} \quad \text{ug/kg} = \frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)(D)}$$

Where:

A_x, I_s, A_{is} = same as for water, above
V_t = Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made)
V_i = Volume of extract added (uL) for purging
D = $\frac{100 - \%\ \text{moisture}}{100}$
W_s = Weight of sample extracted (g) or purged

5.3.12.4 An estimated concentration for non-TCL components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interference shall be used.

The formula for calculating concentrations is the same as above. Total area counts (or peak

heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

6.0 QUALITY CONTROL (All forms can be found in the Appendix)

6.1 Tuning

6.1.1 The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant tune. The time period ends after twelve (12) hours has elapsed according to the system clock.

6.1.2 Tune to meet criteria as shown in Section 5.3.4.7.

NOTE: All instrument conditions must be identical to those used in sample analysis, except that a different temperature program may be used.

6.1.3 BFB criteria MUST be met before any standards, samples, or blanks are analyzed. For CLP, any samples analyzed when tuning criteria have not been met may require reanalysis at no cost to the agency.

6.1.4 Complete Form V when analysis is complete.

6.2 Calibration

6.2.1 Prior to the analysis of samples and required blanks and after turning criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing TCL compound standards. Once the system has been calibrated, the

calibration must be verified each twelve (12) hour time period for each GC/MS system.

6.2.2 Initial calibration of volatile TCL compounds is required at 20, 50, 100,. 150 and 200 ug/L. Surrogate and internal standards shall be used with each of the calibration standards. This will result in 100-1000 total ng analyzed. For CLP, if an analyte saturates at the 200 ug/L concentration level, and the GC/MS system is calibrated to achieve a detection sensititive of no less than 5 ug/L, the laboratory must document it on Form VI and in the Case narrative, and attach a quantitation report and RIC. In this instance, the laboratory should calculate the results based on a four-point initial calibration for the specific analyte that saturates. The use of separate calibration methods which reflect the two different low and medium soil/sediment methods is required. Secondary ion quantitation is only allowed when there are sample interferences with the primary ion. If secondary ion quantitation is used, document the reasons in the Case Narrative. Analyze all method blanks and standards under the same conditions as the sample.

6.2.3 The EPA has specified both the concentration levels for initial calibration and has also specified the specific internal standard to be used on a compound-by-compound basis for quantitation (see table below). Establishment of standard calibration procedures is necessary and deviations by the contractor will not be allowed for CLP work.

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TCL ANALYTES ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d5
Chloromethane	2-Butanone	2-Hexanone
Bromomethane	1,1,1-Trichloroethane	4-Methyl-2-Pentanone
Vinyl Chloride	Carbon Tetrachloride	Tetrachloroethene
Chloroethane	Vinyl Acetate	1,1,2,2-Tetrachloroethane
Methylene Chloride	Bromodichloromethane	Toluene
Acetone	1,2-Dichloropropane	Chlorobenzene
Carbon Disulfide	trans-1,3-Dichloropropene	Ethylbenzene
1,1-Dichloroethene	Trichloroethene	Styrene
1,1-Dichloroethane	Dibromochloromethane	Xylene (total)
1,2-Dichloroethene (total)	1,1,2-Trichloroethane	Bromofluorobenzene (surr)
Chloroform	Benzene	Toluene-d_8 (surr)
1,2-Dichloroethane	cis-1,3-Dichloropropene	
1,2-Dichloroethane-d4 (surr)	Bromoform	

6.2.4 System Performance Check Compounds (SPCC): For volatiles, the five System Performance Check Compounds (SPCCs) are: chloromethane, 1,1,-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane and chlorobenzene. The minimum acceptable average relative response factor (RRF) for these compounds is 0.300, except 0.250 for Bromoform. These compounds typically have RRF's of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines of active sites in the system. For instance:

- Chloromethane - this compound is the most likely compound to be lost if the purge flow is too fast.
- Bromoform - this compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly effected by the tuning of BFB at ions a/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

° Tetrachloroethane, 1,1-Dichloroethane - these compounds can be deteriorated by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

The initial calibration is valid only after both the %RSD for CCC compounds and the minimum RRF for SPCC have been met. Only after both these criteria are met can sample analysis begin.

Once the initial calibration is validated, calculate and report the average relative response factor (RRF) and percent relative standard deviation (%RSD) for all TCL compounds. The analyst shall complete and submit Form V (the GC/MS tune for the initial calibration) and Form VI (Initial Calibration Data) for each instrument used to analyze samples under this protocol.

A calibration standard(s) containing all volatile TCL compounds, including all required surrogates, must be analyzed each twelve hours during analysis. Compare the relative response factor data from the standards each twelve hours with the average relative response factor from the initial calibration for a specific instrument. A system performance check must be made each twelve hours. If the SPCC system criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration (Form VI). If the minimum relative response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

6.2.5 Calibration Check Compounds (CCC) - After the system performance check is met, Calibration Check Compounds listed in the following table are used to check the validity of the initial calibration. Calculate the percent difference using Equation 2.3.

$$\% \text{ Difference} = \frac{\overline{RRF}_I - RRF_c}{\overline{RRF}_I} \times 100 \qquad \text{Eq. 2.3}$$

where:

$\overline{RRF}_I$ = average relative response factor from initial calibration

RRF_c = relative response factor from current calibration check standard

VOLATILE CALIBRATION CHECK COMPOUNDS

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

If the percent difference for any compound is greater than 20%, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than or equal to 25.0%, the initial calibration is assumed to be valid. If the criteria are not met (>25.0% difference), for any one calibration check compound, corrective action MUST be taken. Problems similar to those listed under SPCC could affect this criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration MUST be generated. These criteria MUST be met before sample analysis begins.

The concentration for each volatile TCL compound in the continuing calibration standard(s) is 50 ug/L.

For CLP, the analyst shall complete and submit a Form VII for each GC/MS system utilized for each twelve hour time period. Calculate and report the relative response factor and percent difference (%D) for all compounds. Ensure that the minimum RRF for volatile SPCC's is 0.300 and 0.250 for Bromoform. The percent difference (%D) for each CCC compound must be less than or equal to 25.0 percent.

6.3 Method Blanks

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical scheme. The method blank volume or weight must be approximately equal to the sample volumes or sample weights being processed.

6.3.1 Method blank analysis must be performed at the following frequency: For the analysis of volatile TCL compounds, a method blank analysis must be performed once for each 12-hour time period.

6.3.2 CLP Blanks: A method blank for volatile analysis must contain less than or equal to five times (5X) the Contract Required Quantitation Limit (CRQL from Exhibit C) of methylene chloride, acetone, toluene, and 2-butanone.

For all other TCL compounds not listed above, the method blank must contain less than or equal to the Contract Required Quantitation Limit of any single TCL analyte.

If a laboratory method blank exceeds these criteria, the contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be reextracted/repurged and reanalyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the Case Narrative.

Results of method blank analysis are reported using the Organic Analysis Data Sheet (Form I) and the form for tentatively identified compounds (Form I, TIC). In addition, the samples associated with each method blank must be summarized on Form IV (Method Blank Summary).

ALL sample concentration data as will be reported UNCORRECTED for blanks.

6.4 Surrogates

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.

$$\% \text{ Surrogate Recovery} = \frac{Q_d}{Q_a} \times 100$$

where: Q_d = quantity determined by analysis
Q_a = quantity added to sample

6.4.1 Surrogate Recovery Limits are as follows:

REQUIRED SURROGATE SPIKE RECOVERY LIMITS

Fraction	Surrogate Compound	Water	Low/Medium Soil
VOA	Toluene-d_8	88-110	81-117
VOA	4-Bromofluorobenzene	86-115	74-121
VOA	1,2-Dichloroethane-d_4	76-114	70-121

6.4.2 Method Blank Surrogate Spike Recovery: The laboratory must take the actions listed below if recovery of any one surrogate compound in the volatiles fraction of the method blank is outside of the required surrogate spike recovery limits.

- Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc; also check instrument performance.
- Reanalyze the blank or extract if steps above fail to reveal the cause of the noncompliant surrogate recoveries.

- For CLP, if the blank is a methanol extract for medium level soil samples, reextract and reanalyze the blank if reanalysis alone fails to reveal the cause of the noncomplaint surrogate recoveries.

  If all measures above fail to correct the problem, the analytical system must be considered out of control. The problem MUST be corrected before continuing.

  This may mean recalibrating the instrumentation but it may also mean more extensive action. The specific corrective action is left up to the GC/MS operator. For CLP, when surrogate recovery(ies) in the blank is outside of the contract required windows, all samples associated with that blank MUST be reanalyzed at no additional cost to the Agency.

6.4.3 Sample Surrogate Spike Recovery: The laboratory must take the actions listed below if recovery of any one surrogate compound in the volatiles fraction of the sample is outside of the contract surrogate spike recovery limits. For CLP, the laboratory shall document (in this instance, document means to write down and discuss the problem and corrective action taken in the Case Narrative) deviations outside of acceptable quality control limits by taking the following actions:

- Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also check instrument performance.

- If the above steps fail to reveal a problem, then reanalyze the sample or extract. If reanalysis of the sample or extract solves the problem, then the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the contract windows. This shall be

considered the initial analysis and shall be reported as such on all data deliverables.

- If the sample was a soil extracted with methanol and the steps above fail to solve the problem, then re-extract and reanalyze the sample. If the reextraction and reanalysis solves the problem, then the problem was instrumental. Only submit data from the extraction and analysis with surrogate spike recoveries within the contract windows. This is considered the initial analysis and will be reported as such on all data deliverables.

- If the reextraction and/or reanalysis of the sample does not solve the problem; i.e., surrogate recoveries are outside the contract windows for both analyses, then submit the surrogate spike recovery data and the sample data from both analyses. Full data packages from both analyses must be submitted if we are to be paid for both. Distinguish between the initial analysis and the reanalysis on all data deliverables.

- If the sample with surrogate recoveries outside the limits is the sample used for the matrix spike and matrix spike duplicate, and the surrogate recoveries of the matrix spike and matrix spike duplicate show the same pattern (i.e., outside the limits), then the sample, matrix spike, and matrix spike duplicate do not require reanalysis. Document in the narrative the similarity in surrogate recoveries.

6.4.4 Surrogate Recovery Documentation: The analyst is required to report surrogate recovery data for the following:

° Method Blank Analysis
° Sample Analysis
° Matrix Spike/Matrix Spike Duplicate Analyses
° All sample reanalyses that substantiate a matrix effect

Summarize the surrogate spike recovery data on the Surrogate Spike Percent Recovery Summary (Form III). Method blank surrogate recoveries are plotted on contorl charts.

6.5 Matrix Spike/Matrix Spike Duplicate Analysis (MS/MSD)

6.5.1 Frequency of Analysis

- Each case of field samples received, or
- Each 20 field samples in a case, or
- Each group of samples of a similar concentration level (soils only), or
- Each sample matrix
- Each 14 calendar day period during which samples in a case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group),

whichever is most frequent.

6.5.2 Matrix Spike % Recovery

$$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

MATRIX SPIKE RECOVERY LIMITS (ADVISORY ONLY)

Fraction	Matrix Spike Compound	Water	Soil/Sediment
VOA	1,1-Dichloroethene	61-145	59-172
VOA	Trichloroethene	71-120	62-137
VOA	Chlorobenzene	75-130	60-133
VOA	Toluene	76-125	59-139
VOA	Benzene	76-127	66-142

6.5.3 Relative Percent Difference (RPD)

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where,

RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

6.5.4 Documentation of MS/MSD: The matrix spike (MS) results (concentrations) for volatile TCL compounds shall be reported on Form I (Organic Analysis Data Sheet) and the matrix spike percent recoveries shall be summarized on Form III (MS/MSD Recovery).

The results for volatile TCL compounds in the matrix spike duplicate (MSD) analysis shall be reported on Form I (Organic Analysis Data Sheet) and the percent recovery and the relative percent difference shall be summarized on Form III (MS/MSD Recovery).

MS/MSD recoveries and RPDs are plotted on control charts.

APPENDIX

FORMS

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

Column: (pack/cap) _____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
74-87-3	Chloromethane		
74-83-9	Bromomethane		
75-01-4	Vinyl Chloride		
75-00-3	Chloroethane		
75-09-2	Methylene Chloride		
67-64-1	Acetone		
75-15-0	Carbon Disulfide		
75-35-4	1,1-Dichloroethene		
75-34-3	1,1-Dichloroethane		
540-59-0	1,2-Dichloroethene (total)		
67-66-3	Chloroform		
107-06-2	1,2-Dichloroethane		
78-93-3	2-Butanone		
71-55-6	1,1,1-Trichloroethane		
56-23-5	Carbon Tetrachloride		
108-05-4	Vinyl Acetate		
75-27-4	Bromodichloromethane		
78-87-5	1,2-Dichloropropane		
10061-01-5	cis-1,3-Dichloropropene		
79-01-6	Trichloroethene		
124-48-1	Dibromochloromethane		
79-00-5	1,1,2-Trichloroethane		
71-43-2	Benzene		
10061-02-6	trans-1,3-Dichloropropene		
75-25-2	Bromoform		
108-10-1	4-Methyl-2-Pentanone		
591-78-6	2-Hexanone		
127-18-4	Tetrachloroethene		
79-34-5	1,1,2,2-Tetrachloroethane		
108-88-3	Toluene		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
100-42-5	Styrene		
1330-20-7	Xylene (total)		

1E

VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water)_____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

Column: (pack/cap) _____ Dilution Factor: _____

Number TICs found: _____ CONCENTRATION UNITS: (ug/L or ug/Kg)_____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					
30.					

2A
WATER VOLATILE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

		QC LIMITS
S1 (TOL)	= Toluene-d8	(88-110)
S2 (BFB)	= Bromofluorobenzene	(86-115)
S3 (DCE)	= 1,2-Dichloroethane-d4	(76-114)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogates diluted out

2B
SOIL VOLATILE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Level: (low/med) _____

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

	QC LIMITS
S1 (TOL) = Toluene-d8	(81-117)
S2 (BFB) = Bromofluorobenzene	(74-121)
S3 (DCE) = 1,2-Dichloroethane-d4	(70-121)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogates diluted out

3A

WATER VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMITS REC.
1,1-Dichloroethene					61-145
Trichloroethene					71-120
Benzene					76-127
Toluene					76-125
Chlorobenzene					75-130

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
1,1-Dichloroethene					14	61-145
Trichloroethene					14	71-120
Benzene					11	76-127
Toluene					13	76-125
Chlorobenzene					13	75-130

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

_____

3B

SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level:(low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
1,1-Dichloroethene					59-172
Trichloroethene					62-137
Benzene					66-142
Toluene					59-139
Chlorobenzene					60-133

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
1,1-Dichloroethene					22	59-172
Trichloroethene					24	62-137
Benzene					21	66-142
Toluene					21	59-139
Chlorobenzene					21	60-133

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

4A
VOLATILE METHOD BLANK SUMMARY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Date Analyzed: _____ Time Analyzed: _____

Matrix: (soil/water) _____ Level:(low/med) _____

Instrument ID: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

5A
VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID: _____ BFB Injection Date:_____

Instrument ID: _____ BFB Injection Time:_____

Matrix:(soil/water) _____ Level:(low/med) _____ Column:(pack/cap) _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( )1
174	Greater than 50.0% of mass 95	
175	5.0 - 9.0% of mass 174	( )1
176	Greater than 95.0%, but less than 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1-Value is % mass 174 2-Value is % mass 176

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

6A

VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s):_____ _____

Matrix:(soil/water) _____ Level:(low/med) _____ Column:(pack/cap) _____

Min $\overline{RRF}$ for SPCC(#) = 0.300 (0.250 for Bromoform) Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF20 =_____ RRF50 =_____
RRF100=_____ RRF150=_____ RRF200=_____

COMPOUND	RRF20	RRF50	RRF100	RRF150	RRF200	$\overline{RRF}$	% RSD
Chloromethane	#						#
Bromomethane							
Vinyl Chloride	*						*
Chloroethane							
Methylene Chloride							
Acetone							
Carbon Disulfide							
1,1-Dichloroethene	*						*
1,1-Dichloroethane	#						#
1,2-Dichloroethene (total)							
Chloroform	*						*
1,2-Dichloroethane							
2-Butanone							
1,1,1-Trichloroethane							
Carbon Tetrachloride							
Vinyl Acetate							
Bromodichloromethane							
1,2-Dichloropropane	*						*
cis-1,3-Dichloropropene							
Trichloroethene							
Dibromochloromethane							
1,1,2-Trichloroethane							
Benzene							
trans-1,3-Dichloropropene							
Bromoform	#						#
4-Methyl-2-Pentanone							
2-Hexanone							
Tetrachloroethene							
1,1,2,2-Tetrachloroethane	#						#
Toluene	*						*
Chlorobenzene	#						#
Ethylbenzene	*						*
Styrene							
Xylene (total)							
Toluene-d8							
Bromofluorobenzene							
1,2-Dichloroethane-d4							

7A

VOLATILE CONTINUING CALIBRATION CHECK

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date:_____ Time:_____

Lab File ID: _____ Init. Calib. Date(s):_____ _____

Matrix:(soil/water) _____ Level:(low/med) _____ Column:(pack/cap) _____

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(*) = 25.0%

COMPOUND	$\overline{RRF}$	RRF50	%D
Chloromethane			
Bromomethane			
Vinyl Chloride			
Chloroethane			
Methylene Chloride			
Acetone			
Carbon Disulfide			
1,1-Dichloroethene			
1,1-Dichloroethane			
1,2-Dichloroethene (total)			
Chloroform			
1,2-Dichloroethane			
2-Butanone			
1,1,1-Trichloroethane			
Carbon Tetrachloride			
Vinyl Acetate			
Bromodichloromethane			
1,2-Dichloropropane			
cis-1,3-Dichloropropene			
Trichloroethene			
Dibromochloromethane			
1,1,2-Trichloroethane			
Benzene			
trans-1,3-Dichloropropene			
Bromoform			
4-Methyl-2-Pentanone			
2-Hexanone			
Tetrachloroethene			
1,1,2,2-Tetrachloroethane			
Toluene			
Chlorobenzene			
Ethylbenzene			
Styrene			
Xylene (total)			
Toluene-d8			
Bromofluorobenzene			
1,2-Dichloroethane-d4			

8A
VOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab File ID (Standard): _____ Date Analyzed:_____

Instrument ID: _____ Time Analyzed:_____

Matrix:(soil/water) _____ Level:(low/med) _____ Column:(pack/cap) _____

	IS1(BCM) AREA #	RT	IS2(DFB) AREA #	RT	IS3(CBZ) AREA #	RT
12 HOUR STD						
UPPER LIMIT						
LOWER LIMIT						
EPA SAMPLE NO.						
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (CBZ) = Chlorobenzene-d5

UPPER LIMIT = + 100% of internal standard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

PEI ASSOCIATES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

SOP No. 10-001-01

Approved by	Date

Terminated by	Date

Title:	Word Processing Disk Number:
pH Analysis	1850

Prepared by	Approved by / Date	QA Concurrence / Date
[signature]	[signature] 2/3/88	Lauren Drees 2/9/88

1.0 PURPOSE

To describe the procedure for determining sample pH.

2.0 APPLICATION

This procedure is applicable to waters, wastes, and soils.

3.0 REFERENCES

3.1 EPA Chemical Analysis of Water and Wastes 1983, Method 150.1.

3.2 EPA SW-846 3rd ed., Methods 9040 and 9045.

4.0 ASSOCIATED SOPs

4.1 SOP No. 06-005-00, pH Meter Calibration.

5.0 PROCEDURE

5.1 Equipment

5.1.1 pH meter, Sargent Welch Model LSX

5.1.2 Beakers, 50 ml.

5.2 pH Analysis of Water Samples (or wastes where the aqueous phase constitutes at least 20% of the total volume).

5.2.1 Allow the samples to come to room temperature.

5.2.2 Place approximately 30 ml of sample in a 50 ml beaker. Add a small stirring bar and place the beaker on a stir plate.

5.2.3 Lower the electrode into the sample. Make sure it does not touch the bottom or sides of the beaker. Begin slowly mixing (mix all buffers and samples at the same speed).

5.2.4 Switch the OPERATION knob to pH/ION. When the reading has stabilized, record the pH.

5.2.5 Turn the OPERATION knob to STANDBY.

5.2.6 Remove the electrode from the sample, rinse with deionized water and blot dry.

5.2.7 Repeat Steps 5.2.1-5.2.7 for each sample.

5.3 pH analysis of soils

5.3.1 To 20 g of soil in a 50 ml beaker, add 20 ml of deionized water. Stir the suspension several times during the next 30 minutes.

5.3.2 Let the soil suspension stand for about one hour to allow most of the suspended matter to settle.

5.3.3 Lower the electrode into the beaker until the tip is submerged in the supernatant layer.

5.3.4 Turn the OPERATION knob to pH/ION. Allow the reading to stabilize and record the pH.

5.3.5 Turn the OPERATION Knob to STANDBY.

5.3.6 Remove the electrode from the sample, rinse with deionized water and blot dry.

5.3.7 Repeat Steps 5.3.1-5.3.6 for each sample.

Note: Keep the tip of the electrode submerged in the pH=7 buffer when not in use.

6.0 Quality Control

6.1 Analyze duplicates at a frequency of 10%.

SOP

Standard Operating Procedure

SOP No: 07-019-00
Date Initiated: 5/16/88
Page 1 of 4
Date Revised:

itle:

Chloride by Mercuric Nitrate Titration

Word Processing Disk Number:

1935

epared by	Approved by	Date	QA Concurrence	Date
Vo Lewis	Cyprie Bearden	6/3/88	Lauren Drees	6/3/88

1.0 PURPOSE

To describe the procedure for the determination of chloride by titration with mercuric nitrate.

2.0 APPLICATION

This procedure is applicable to waters, wastewaters and ground waters.

3.0 REFERENCES

3.1 US EPA Methods for Chemical Analysis of Water and Wastes, 1983, Method 325.3.

3.2 US EPA SW-846, 3rd edition, Method 9252.

3.3 Standard Methods for the Examination of Water and Wastewater, 16th edition, Method 407B.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 Sodium chloride standard, 0.141 N: Dissolve 8.2414 g NaCl in deionized water and dilute to one liter. Shelf-life = 1 year.

5.1.2 Sodium chloride standard, 0.0141 N: Dilute 50 ml of 0.141 N NaCl standard to 500 ml with deionized water. Shelf-life = 1 year.

5.1.3 Mercuric nitrate titrant, ~0.141 N: Dissolve 25 g $Hg(NO_3)_2 \cdot H_2O$ in 900 ml deionized water acidified with 5 ml conc. HNO_3 and dilute to one liter. Store in a dark bottle.

5.1.4 Mercuric nitrate titrant, ~0.0141 N: Dilute 50 ml of 0.141 N $Hg(NO_3)_2$ to 500 ml with deionized water. Store in a dark bottle.

5.1.5 Mixed indicator: Dissolve 0.5 g crystalline diphenylcarbazone and 0.05 g bromophenol blue powder in 75 ml of 95% ethanol. Dilute to 100 ml with 95% ethanol. Store in brown bottle and discard after 6 months.

5.1.6 Indicator-acidifier reagent: Dissolve, in the order named, 250 mg s-diphenylcarbazone, 4.0 ml conc. HNO_3, and 30 mg xylene cyanol FF in 100 ml of 95% isopropanol. Store in a dark bottle in a refrigerator. Shelf-life = one month.

5.1.7 Nitric acid, HNO_3, 0.1 N: Dilute 6.25 ml conc. HNO_3 to one liter with deionized water.

5.1.8 Sodium hydroxide, NaOH, 0.1 N: Dissolve 4 g NaOH in deionized water and dilute to one liter.

5.1.9 Erlenmeyer flasks, 250 ml.

5.1.10 Buret with 0.1 ml graduation intervals.

5.2 Titration of Chloride Concentrations Less than 100 mg/l

5.2.1 Standardization of ~0.0141 N $Hg(NO_3)_2$

Pipet three 20 ml aliquots of the 0.0141 N NaCl standard into 250 ml Erlenmeyer flasks and dilute to 100 ml with

deionized water. Add 1.0 ml of indicator-acidifier reagent (the color should be green-blue at this point).

Titrate with ~0.0141 N $Hg(NO_3)_2$ to the first permanent dark purple. The solution turns from green-blue to blue a few drops before the end point

$$\text{Normality of } Hg(NO_3)_2 = \frac{\text{ml NaCl std} \times 0.0141}{\text{ml } Hg(NO_3)_2 \text{ used}}$$

Average the normalities of the 3 determinations.

5.2.2 Sample Analysis

Use 100 ml sample or an aliquot diluted to 100 ml with deionized water. Add 1.0 ml of indicator-acifier reagent (The color of the solution should be green-blue at this point). A light green indicates pH less than 2.0; a pure blue indicates pH more than 3.8. For highly alkaline or acid waters, adjust pH to about 8 before adding the indicator-acidifier reagent.

Titrate with $Hg(NO_3)_2$ titrant to a definite purple end point. The solution turns from green-blue to blue a few drops before the endpoint.

Analyze a blank by titrating 100 ml deionized water using the same procedure.

5.3 Titration of Chloride Concentrations Greater Than 100 mg/l

5.3.1 Standardization of ~0.141 N $Hg(NO_3)_2$

Pipet three 5.0 ml aliquots of the 0.141 N NaCl standard into Erlenmeyer flasks and dilute to 50 ml with deionized water. Add 0.5 ml mixed indicator reagent and mix well. The color should be purple. Add 0.1 N HNO_3 dropwise until the color turns just yellow. Titrate with ~0.141 N Hg $(NO_3)_2$ titrant to the first permanent dark purple.

$$\text{Normality of Hg } (NO_3)_2 = \frac{\text{ml NaCl std} \times 0.141}{\text{ml } Hg(NO_3)_2 \text{ used}}$$

Average the normalities of the 3 determinations.

5.3.2 Sample Analysis

Use a sample portion (up to 50 ml) requiring less than 5 ml titrant to reach the end point. Dilute to 50 ml with deionized water if necessary. Add 0.5 ml mixed indicator reagent and mix well. The color should be purple. Add 0.1 N HNO_3 dropwise until the color turns just yellow. Titrate with $Hg(NO_3)_2$ titrant to the first permanent dark purple. Analyze a blank by titrating 50 ml deionized water using the same procedure.

5.4 Calculations

$$\text{mg/l chloride} = \frac{(A-B) \times N \times 35{,}450}{\text{ml sample}}$$

where A = ml titration for sample
B = ml titration for blank
N = normality of $Hg(NO_3)_2$

6.0 QUALITY CONTROL

6.1 Analyze a blank (as described in the method) with each sample batch.

6.2 Analyze an SRS with each sample batch. Plot recovery on the appropriate control chart.

6.3 Analyze an MS/MSD pair for every 20 samples. For samples <100 mg/l, add 1.0 ml of 1000 mg/l Cl solution. For samples >100 mg/l add 5.0 ml of 1000 mg/l Cl solution.

PEI ASSOCIATES, INC.
SOP CONTINUING APPROVAL/TERMINATION FORM

SOP No. 07-019-00

Approved by	Date

Terminated by	Date

SOP

Standard Operating Procedure

SOP No: 01-007-00
Date Initiated: 3/21/88
Page 1 of 3
Date Revised:

Title:

Inorganic Glassware Cleaning

Word Processing Disk Number:

1892

Prepared by	Approved by	Date	QA Concurrence	Date
Jim McCarthy	Cyprian Bender	3/17/88	Lauren Drees	3/21/88

1.0 PURPOSE

This procedure describes the preparation and cleaning of glassware to be used in the inorganic laboratory.

2.0 APPLICATION

This procedure is applicable to all glassware, including pipettes.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Equipment/materials

5.1.1 Glassware cleaning detergent containing no phosphates and having a neutral pH (liquid Alconox or equivalent).

5.1.2 Glassware cleaning brushes of varying sizes and dimensions relative to the glassware being cleaned.

5.1.3 Drying oven.

5.1.4 Safety equipment (i.e., lab coat, gloves with chemical resistance, safety glasses).

5.1.5 Wash basin or sink with drain plug.

5.1.6 Chromerge solution: Add 1 vial Chromerge to one bottle H_2SO_4 and mix.

5.1.7 Pipette washer.

5.1.8 Pipette washing solution: Fill pipette washer about 1/2 full with ice. Add 3 bottles H_2SO_4 and 2 packages of Nochromix. Mix thoroughly.

5.2 Glassware Cleaning

5.2.1 Wash glassware in a hot detergent/water solution (use detergent: water concentration recommended on detergent label) with appropriate cleaning brush until no visible trace of particles or contamination exists.

5.2.2 After soapy water wash, rinse glassware immediately with hot tap water until all traces of soap are gone.[1]

5.2.3 Rinse glassware thoroughly with deionized water.

5.2.4 Place glassware in an 80°C drying oven until completely dry.

5.2.5 Glassware should be stored in cabinets or, if necessary, in a location free of contaminants which may interfere with a specific analysis.

Notes: 1) If soap and water alone do not remove all residue, soak glassware in Chromerge solution.

2) Some glassware may need additional cleaning as specified in a particular method.

5.3 Pipette Cleaning

5.3.1 Rinse pipettes with deionized water after use.

5.3.2 Place pipettes (tip up) in the pipette washer.

[1] If glassware is used for metals analyses, rinse with 10 percent HCl solution.

5.3.3 When full, connect washer to tap water and allow to cycle several times.

5.3.4 Connect washer to deionized water and allow to cycle several times. Let drain.

5.3.5 Place clean pipettes in a rack or drawer, as appropriate.

5.3.6 Replace pipette wash solution when necessary (when pipettes no longer appear to be getting clean).

SOP

Standard Operating Procedure

SOP No: 01-002-00
Date Initiated: 1/18/88
Page 1 of 2
Date Revised:

itle:

Organic Glassware Cleaning

Word Processing Disk Number: 1740

epared by	Approved by	Date	QA Concurrence	Date
Thomas Boyd	[signature]	1/27/88	Lauren Drees	1/13/88

1.0 PURPOSE

To describe the procedure for the cleaning and preparation of glassware used in the organic laboratory.

2.0 APPLICATION

This procedure applies to glassware used for all organic analyses, including ZHE.

3.0 REFERENCES

None

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURES

5.1 Remove surface residuals immediately after use by rinsing glassware with deionized water.

5.2 Wash glassware in a hot solution of cleaning reagent (liquid Alconex, Micro or equivalent) and tap water. If necessary, remove any marker with acetone.

5.3 Rinse with hot tap water to flush away flotated residue.

5.4 Rinse with deionized water to remove metallic deposits from the tap water.

5.5 Rinse with methanol (distilled in glass grade).

5.6 Rinse with methylene chloride (distilled in glass grade).

5.7 Rinse with methanol again.

5.8 Place glassware in an oven to dry.

NOTE: For glassware to be used for ZHE analysis, omit the solvent rinsing (Steps 5.5, 5.6, 5.7).

	SOP *Standard Operating Procedure*	SOP No: 07-005-00 Date Initiated: 1/1/88 Page 1 of 3 Date Revised:

Title: Total Dissolved Solids (TDS)	Word Processing Disk Number: 1740

Prepared by	Approved by	Date	QA Concurrence	Date
Frank L. Stephens	Eugene Brandon	12/11/87	Lauren Dreer	12/11/87

1.0 PURPOSE

To describe the procedure used for the determination of total dissolved solids (filterable residue).

2.0 APPLICATION

This procedure applies to any aqueous sample which can be filtered through a glass fiber filter.

3.0 REFERENCES

3.1 EPA Chemical Analysis of Water and Wastes, 1983, Method 160.1.

4.0 ASSOCIATED SOPs

4.1 SOP No. 07-004-00, Total Suspended Solids (TSS).

5.0 PROCEDURE

5.1 Equipment

5.1.1 Beakers, 250 ml.

5.1.2 Filter flask and Buchner funnel with vacuum source.

5.1.3 Analytical balance.

5.1.4 Desiccator.

5.1.5 Drying oven, 180°C.

5.1.6 Glass fiber filters, 7.0 cm diameter.

5.2 Filter Preparation

5.2.1 Place stacks of approximately 25 filters in the funnel and rinse with three successive 100 ml portions of deionized water.

5.2.2 Separate filters, place in a porcelain crucible and dry for at least one hour at 103°C.

5.2.3 Desiccate filters until needed.

5.3 Beaker Preparation

5.3.1 Heat clean 250 ml beakers in oven at 180°C for one hour.

5.3.2 Desiccate until needed.

5.4 Analysis

5.4.1 Weigh beakers needed on an analytical balance and record weight.

5.4.2 Place filter in filtering apparatus, turn on vacuum and rinse with a small amount of deionized water (to create good filter-funnel seal).

5.4.3 Filter 100 ml of well mixed sample through a filter (use less if necessary). Rinse with three successive 10 ml portions of deionized water. Continue applying vacuum until all traces of water have passed through the filter,

5.4.4 Turn off vacuum source and transfer filtrate to the tared beaker. Rinse flask into beaker. Place beaker in 180°C oven overnight (or until dry).

5.4.5 If suspended solids are to be determined also, the filter can now be transferred (see SOP No. 07-004-00, Total Suspended Solids (TSS)).

5.4.6 Desiccate beakers for at least 30 minutes and then weigh.

5.4.7 If the total residue in the beaker exceeds 0.5 g repeat the analysis using a smaller aliquot.

5.5 Calculations

$$\text{TDS, mg/l} = \frac{(A-B) \times 1000}{C}$$

where:

A = weight of beaker + residue, mg
B = weight of beaker, mg
C = ml of sample filtered

6.0 Quality Control

6.1 A minimum of one SRS must be run with each sample set.

6.2 Duplicates must be analyzed at a frequency of 10%.

pel	**SOP** *Standard Operating Procedure*	SOP No: 07-009-00 Date Initiated: 1/1/88 Page 1 of 3 Date Revised:

Title:	Word Processing Disk Number:
Total and Bicarbonate Alkalinity	1740

Prepared by	Approved by	Date	QA Concurrence	Date
[signature]	[signature]	1/1/88	[signature]	1/1/88

1.0 PURPOSE

To describe the procedure for the determination of the alkalinity, or sum of the titrable bases, in a given water sample. From this information, an indication of the concentration of carbonate, bicarbonate and hydroxide constituents can be obtained.

2.0 APPLICATION

This procedure can be applied to any water or wastewater sample. The sample must be at room temperature before analysis is begun.

3.0 REFERENCES

3.1 EPA Methods for Chemical Analysis of Water and Wastes, 1983, Method 310.1.

3.2 Standard Methods for the Analysis of Water and Wastewater, 16th edition, Method 403.

ASSOCIATED SOPs

4.1 SOP No. 06-005-00 pH Meter Calibration.

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 THAM (Tris(hydroxymethyl)aminomethane)

5.1.2 Sulfuric acid titrant ~0.03 N: Dilute 2 ml of concentrated H_2SO_4 to 2000 ml with deionized water.

5.1.3 pH meter.

5.2 Standardization of acid

5.2.1 Weigh three ~40 mg portions of THAM to the nearest 0.1 mg into 125 ml Erlenmeyer flasks.

5.2.2 Add deionized water to bring the volume to about 50 ml and let dissolve.

5.2.3 Titrate with sulfuric acid titrant using methyl orange as the indicator (color change from yellow to orange).

$$\text{Normality of acid} = \frac{\text{mg THAM}}{\text{ml titrant} \times 121.14}$$

5.3 Calibration of pH meter - see SOP No. 06-005-00

5.4 Analysis

5.4.1 Total alkalinity - Measure 100 ml of sample at room temperature into a 250 ml beaker and stir gently. (Use less sample if necessary but do not dilute.) Insert pH electrode and record pH. Titrate with sulfuric acid titrant to a pH of 4.5. Record the ml titrant.

$$\text{total alkalinity, mg/l } CaCO_3 = \frac{T \times N \times 50{,}000}{\text{sample vol., ml}}$$

where T = total ml titrant
N = normality of sulfuric acid titrant

5.4.2 Bicarbonate alkalinity - Same as for total alkalinity except the ml titrant to pH 8.3 must be recorded as well as the total ml titrant to 4.5.

$$\text{Bicarbonate alkalinity, mg/l } CaCO_3 = \frac{(T-2P) \times N \times 50{,}000}{\text{sample vol., ml}}$$

where T = total ml titrant
P = ml titrant to 8.3
N = normality of sulfuric acid titrant

Note: If P = 0 (sample pH is below 8.3), the bicarbonate alkalinity is equal to the total alkalinity.

6.0 QUALITY CONTROL

6.1 Analyze a minimum of one SRS with each set.

6.2 Analyze duplicate samples at a frequency of 10%.

SOP

Standard Operating Procedure

SOP No: 07-023-00
Date Initiated: 5/30/88
Page 1 of 3
Date Revised:

tle:

Total Hardness

Word Processing Disk Number:

1935

epared by	Approved by	Date	QA Concurrence	Date
Grace Plemmons	Ken Mueller	8/31/88	Lauren Dreier	8/31/88

1.0 PURPOSE

To describe the procedure for determining calcium and magnesium ion concentration, or hardness (mg/l as $CaCO_3$).

2.0 APPLICATION

This method is applicable to waters and wastewaters.

3.0 REFERENCES

US EPA Methods for Chemical Analysis of Water and Wastes, Method 130.2.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 EDTA titrant, .02 N: Dissolve 3.723 g analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, $Na_2H_2C_{10}H_{12}O_8N_2 \cdot 2H_2O$ in deionized water and dilute to one liter.

5.1.2 Water hardness buffer: Commercially purchased.

5.1.3 Sodium cyanide powder.

5.1.4 NaCl dye mix: Mix together 0.5 g Erichrome Black T and 100 g NaCl.

5.1.5 Standard calcium carbonate solution, .02 N: Place 1.0000 g anhydrous calcium carbonate in a 500 ml flask. Add, a little at a time, 1+1 HCl until all of the $CaCO_3$ has dissolved. Add 200 ml of distilled water. Boil for a few minutes to expel CO_2. Cool. Add a few drops of methyl red indicator and adjust to intermediate orange color by adding 3N NH_4OH or 1+1 HCl. Quantitatively transfer to a liter volumetric flask and dilute to mark.

5.1.6 Hydrochloric acid solution, 1+1.

5.1.7 Ammonium hydroxide solution, 3 N: Dilute 210 ml of conc. NH_4OH to 1 liter with deionized water.

5.2 Standardization of EDTA titrant

5.2.1 Prepare a standard by pipetting 10.0 mls of standard calcium carbonate solution into a 125 ml Erlenmeyer flask and dilute to 50 ml with deionized water.

5.2.2 Add the following in a hood:

1 ml buffer
1 scoop sodium cyanide powder
1/2 scoop of NaCl dye mix

5.2.3 Titrate with EDTA titrant to a blue endpoint.

5.2.4 Repeat 5.2.1-5.2.3 twice more.

5.2.5 $N\ EDTA = \frac{0.2}{ml\ EDTA}$

Average the normalities of the three determinations.

5.3 Sample Analysis

5.3.1 Place 25 ml of sample in a 125 ml Erlenmeyer flask and dilute to 50 mls with deionized water.

5.3.2 Add the following in a hood:

1 ml buffer

1 scoop sodium cyanide

1/2 scoop of NaCl dye mix

5.3.3 Titrate with EDTA titrant to a blue endpoint (color changes from pinkish-red to blue).

Note 1: The sample should require <15 ml EDTA titrant and the titration should be completed within 5 minutes of the buffer addition.

Note 2: For samples with low hardness (<5 mg/l), use a 100 ml aliquot and proportionately larger amounts of buffer, NaCN and dye.

5.4 Calculations

$$\text{Total hardness, mg/l } CaCO_3 = \frac{A \times N \times 50000}{\text{mls sample}}$$

where: A = mls EDTA titrant

N = normality of titrant

6.0 QUALITY CONTROL

6.1 Analyze an SRS with each set of samples. Plot the recovery on a control chart.

6.2 Analyze duplicates at a frequency of 10%. Once enough data is collected, control charts will be used.

SOP

Standard Operating Procedure

SOP No: 07-013-00
Date Initiated: 2/22/88
Page 1 of 3
Date Revised:

Title: Sulfate (Turbidimetric)

Word Processing Disk Number: 1850

Prepared by	Approved by	Date	QA Concurrence	Date
[signature]	[signature]	2/16/88	[signature]	2/16/88

1.0 PURPOSE

To describe the procedure for determining the sulfate (SO_4) concentration of a sample by measuring the turbidity of the sample after precipitation with barium chloride.

2.0 APPLICATION

This procedure is applicable to water, wastewater, and groundwater samples.

3.0 REFERENCES

3.1 U.S. EPA Chemical Analysis of Water and Wastes, 1983, Method 375.4.

3.2 EPA SW-846, 3rd edition, Method 9038.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 Sulfaver IV pillows: Purchase from Hach Chemicals.

5.1.2 Stock sulfate standard, 1000 µg/ml: Dissolve 1.3765 g of oven-dried ammonium sulfate in deionized water and dilute to one liter.

5.1.3 Working sulfate standard, 80 µg/ml: Dilute 8.0 ml of the stock sulfate standard to 100 ml with deionized water.

5.1.4 Spectrophotometer for use at 450 nm.

5.1.5 Spectrophotometer tubes.

5.2 Analysis

5.2.1 Prepare the following standards by pipetting the volumes given into spectrophotometer tubes and diluting to 25 ml.

ml working standard	µg SO_4
0	0
1	80
2	160
5	400
10	800
15	1200
20	1600
25	2000

5.2.2 Pipet 25 ml sample, or an aliquot diluted to 25 ml, into a spectrophotometer tube.

Note: If samples are colored or turbid, read the absorbance now before the Sulfaver pillow is added. The µg value obtained will be subtracted from the value obtained after the addition of the Sulfaver pillow.

5.2.3 Add the contents of one Sulfaver pillow to each tube and mix by inverting several times.

5.2.4 Between 5 and 10 minutes after mixing, read the absorbance at 450 nm. Zero the instrument with deionized water.

5.3 Calculation

Plot the µg SO_4 versus absorbance.

$$\mu g/ml\ SO_4 = \frac{\mu g \text{ from curve}}{ml \text{ sample}}$$

Note: If samples were colored or turbid, then:

$$\mu g/ml\ SO_4 = \frac{\mu g \text{ from curve (after)} - \mu g \text{ from curve (before)}}{ml \text{ sample}}$$

6.0 QUALITY CONTROL

6.1 Analyze an SRS with each sample set. Plot the % recovery on the appropriate control chart to determine if the method was in control.

6.2 Analyze an MS/MSD pair with each set or for every 20 samples, whichever is more frequent.

SOP

Standard Operating Procedure

SOP No: 06-001-00
Date Initiated: 12/1/87
Page 1 of 6
Date Revised:

Title:
Non-Purgeable Organic Carbon (NPOC) in Waters

Word Processing Disk Number:
1740

Prepared by	Approved by	Date	QA Concurrence	Date
Lauren Drees	Cynthia Sundberg	12/1/87	Thomas J. Hayes	12/1/87

1.0 PURPOSE

To define the analytical procedures for the determination of NPOC in water samples.

2.0 SCOPE

Waters are analyzed for NPOC by UV-promoted wet chemical oxidation. Non-purgeable organic carbon is converted to CO_2 which is then measured directly by an infrared detector.

3.0 REFERENCES

3.1 EPA SW-846, 3rd ed., Method 9060.

3.2 EPA Methods for Chemical Analysis of Water and Wastes, 1983, Method 415.1.

3.3 Xertex/Dohrmann's DC-80 Automated Laboratory Total Organic Carbon Analyzer manual.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Reagents/Equipment

5.1.1 Oxygen, 99.9% pure.

5.1.2 Potassium persulfate solution, 2%: Dissolve 20 g of reagent grade $K_2S_2O_8$ in one liter of deionized water. Add 1 ml concentrated phosphoric acid and mix well. Store in a cool dark location. Shelf life is approximately one month.

5.1.3 TOC standard, 2000 ppm: Add 425 mg dried potassium hydrogen phthalate (KHP) to deionized water in a 100 ml volumetric flask. Add 0.1 ml phosphoric acid and bring to volume. Store in dark glass under refrigeration and replace monthly.

5.1.4 TOC standard, 400 ppm: Dilute 20.0 ml of the 2000 ppm standard to 100 ml with deionized water. Store in dark glass under refrigeration and prepare fresh weekly.

5.1.5 TOC standard, 10.0 ppm: Dilute 1.0 ml of the 2000 ppm standard to 200 ml with deionized water. Prepare fresh daily.

5.1.6 Syringe, 1 ml.

5.1.7 Dohrmann Model DC-80 TOC analyzer with autosampler.

5.2 Instrument Set-Up

5.2.1 Fill reactor with potassium persulfate solution.

5.2.2 Connect all tubing as in Figure 1 attached.

5.2.3 Put reagent line in reagent bottle.

5.2.4 Put pressure fingers on.

5.2.5 Turn on in order: POWER, PUMP, LAMP.

5.2.6 Turn gas to 33 psi.

5.2.7 With DETECTOR/PPM switch in detector position, allow baseline to stabilize and adjust to 0.0100.

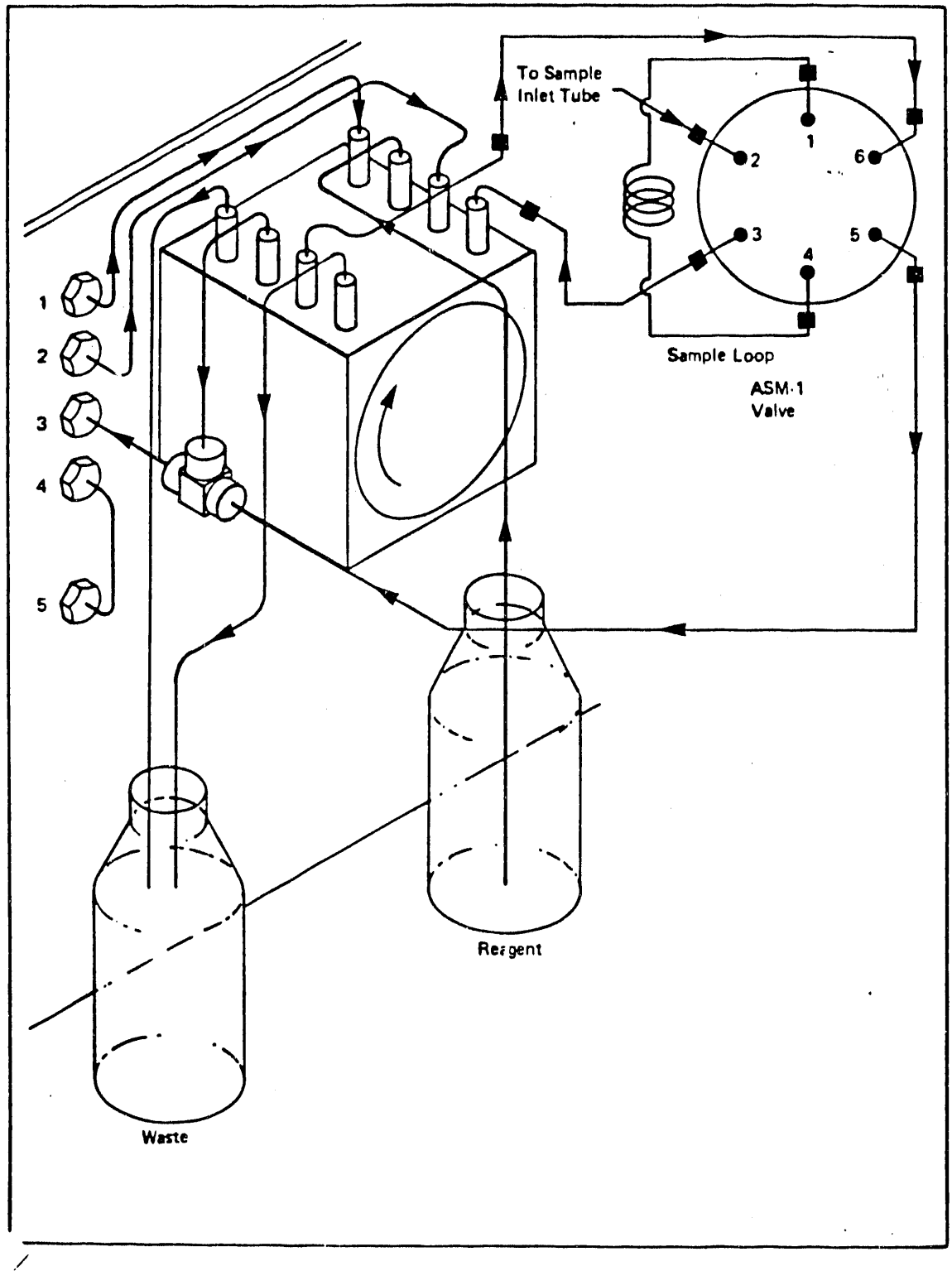

To Sample
Inlet Tube
1
2
3
4
5
6
Sample Loop
ASM-1
Valve
Reagent
Waste

Figure 1

5.2.8 Set DETECTOR/PPM switch to ppm. Set TOC/POC mode switch to TOC.

5.2.9 Remove any previous calibrations by depressing the CALIBRATE button for several seconds. The printer will print "no cal" and the range.

5.3 Calibration

5.3.1 Low-Level TOC: For water samples with an expected TOC level between 0.1-20 mg/l, use the 1000 µl sample range. Calibration is performed using the 10.0 ppm TOC standard. Fill a TOC tube with standard and analyze using the manual mode of the autosampler. Two aliquots of the standard are analyzed. If the values agree within 5%, depress the CALIBRATE button for one second and the instrument is calibrated. If the values do not agree, press the calibration button once and then again to remove the unacceptable calibration and repeat the process.

5.3.2 Mid-Level TOC: For water samples with an expected TOC level between 10-800 mg/l, use the 200 µl sample loop and range. Calibration is performed as described for the Low-Level Method, but using the 400 ppm TOC standard.

5.3.3 High-Level TOC: For water samples with an expected TOC level between 100-4000 mg/l, use the 40 µl loop and range. Calibration is performed as described for the Low-Level Method, but using the 2000 ppm TOC standard.

5.4 Analysis

5.4.1 A system blank (used only in Low-Level TOC analysis) is obtained by withdrawing 1.0 ml of reactor fluid with a syringe through the septum located at the front of the reactor. This is manually injected into the sample port immediately after the green START button has been depressed. Repeat until a consistent blank is obtained.

5.4.2 The sample must be preserved to pH <2 with H_2SO_4 and purged to remove inorganic carbon due to carbonates and bicarbonates. During the removal of inorganic carbon, purgeable organic carbon is also removed. (Note 1) The autosampler is designed to purge the samples before injection into the system. Once tubes are filled and positioned in the tray, automatic operation is used. (Note 2)

mg/l NPOC = mg/l, sample - mg/l, system blank

5.5 Instrument Shutdown

5.5.1 Turn off in order: LAMP, PUMP, POWER.

5.5.2 Remove pressure fingers.

5.5.3 Remove reagent line from reagent bottle.

5.5.4 Turn gas to 5 psi and bleed by pressing Autosampler AUTO button several times.

6.0 QC

6.1 Analyze an SRS as an initial calibration verification standard, after every 10 samples, and at the end of a run.

6.2 Analyze a matrix spike/matrix spike duplicate pair every twenty samples.

Notes: 1) Remember that this analysis actually gives a value for Non-Purgeable Organic Carbon (NPOC). Any volatile organic carbon is removed with the inorganic carbon during purging. To get TOC (Total Organic Carbon), Purgeable Organic Carbon (POC) must also be determined and added to the NPOC value.

2) Sometimes interferences (such as solid material, high chloride) are present which do not allow the sample to be completely chemically oxidized in the time of the run. These problems are not observed in combustion oxidation. Waters

with NPOC concentrations exceeding 20 ppm may also be analyzed by the combustion method as for soil and sludge analyses (SOP 06-002-00 Non-Purgeable Organic Carbon (NPOC) in Soils and Sludges).

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-025-00 Date Initiated: 6/23/89 Page 1 of 5 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance In The Metals Laboratory	2452

Prepared by	Approved by	Date	QA Concurrence	Date
Jeff Armie	Ken Mueller	6/20/89	Janet M. Jones Lauren Drees Linda Reeves	7/20/89 6/20/89 7/21/89

1.0 PURPOSE

To help keep an instrument running properly, routine preventive maintenance must be performed. Maintaining an effective routine preventive maintenance schedule requires proper documentation. The purpose of this SOP is to assist the analyst in keeping an effective, well documented routine preventive maintenance log book.

2.0 APPLICATION

This SOP was written specifically for the Perkin-Elmer Plasma II, 3030B, and 560 Atomic Spectrometers. The scope could be modified for additional instruments used in the metals analyses as they added to the laboratory.

3.0 REFERENCES

3.1 Instructional Manuals for the Plasma II Emission Spectrometer, Book 1, 2, and 3.

3.2 Instructional Manual for the 3030B Atomic Absorption Spectrophotometer.

3.3 Instructional Manual for the 560 Atomic Absorption Spectrophotometer.

4.0 ASSOCIATED SOP's

None.

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Plasma II

Maintenance	Check frequency	Replace frequency	Clean frequency
Pump tubing	Daily	Every 2 days	When needed
Capillary tubing	Daily	When needed	When needed
Quartz tubing	Daily	When needed	When needed
Injector tube	Daily	When needed	When needed
Nebulizer insert	When needed	When needed	When needed
RF coil	Daily	When needed	When needed
Quartz bonnet	Daily	When needed	When needed
Drain bottle	Daily		When needed
Purge gas	Daily	When needed	When needed
Purge windows	Daily	When needed	When needed
Vacuum pump oil	Daily	Every 6 mos.	
Adsorption trap		Every month	
Exhaust filter		Annually	
Nitrogen filter		Annually	

5.1.2 3030B

Maintenance	Check frequency	Replace frequency	Clean frequency
Contact rings	Daily	Every 6 mos.	Daily
Graphite tube	Daily	When needed	
Quartz windows	Daily	When needed	When needed
Injector tubing	Daily	When needed	When needed

5.1.3 560

Maintenance	Check frequency	Replace frequency	Clean frequency
Quartz tube assembly	Daily		When needed
Quartz windows	Daily	When needed	When needed

5.2 Documentation in Logbook

Each day, before an analyst begins to run an instrument, the routine preventative maintenance check sheet must be filled out (Figure 1).

5.2.1 Always begin by entering the date in the top left corner of the check sheet.

5.2.2 After the daily checks are made the analyst enters his or her initials in the appropriate column in the check sheet.

5.2.3 In the event that an item on the check sheet is replaced or cleaned, the analyst's initials are entered in the appropriate column on the check sheet.

5.2.4 The comments column of the check sheet is to be used for referencing detailed accounts of all uncustomary repairs, replacements, and maintenance to the back side of the check sheet (Figure 2) by entering "on back" in the space provided. The analyst's initials and the date are to be entered immediately after any comments are entered on the back page.

5.2.5 A yearly calendar is kept in the log book to help remind the analyst of monthly, semi-annual, and annual maintenance.

5.2.6 Preventive maintenance documentation is reviewed monthly by the Metals Team Leader.

PREVENTIVE/ROUTINE MAINTENANCE

Date:

ICP	Check	Replace	Clean	Comments
Pump Tubing				
Capillary Tubing				
Quartz Tubing				
Injector Tube				
Nebulizer Insert				
RF Coil				
Quartz Bonnet				
Drain Bottle				
Purge Gas				
Purge Windows				
Vacuum Pump Oil				

AA 3030B	Check	Replace	Clean	Comments
Contact Rings				
Graphite Tube				
Quartz Windows				
Injector Tubing				

AA 560	Check	Replace	Clean	Comments
Quartz Tube Assembly				
Quartz Windows				

Figure 1

Documentation of Maintenance/Repairs Performed:

Figure 2

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-024-00 Date Initiated: 6/23/89 Page 1 of 9 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the Gas Chromatography/Mass Spectrometry Lab	2452

Prepared by	Approved by	Date	QA Concurrence	Date
Dwight R. Hayes, Jr.	C. Waldwell	6/29/89	Janet M. Jones Lauren Greco Linda Pierucci	7/20/89 6/29/89 7/21/89

1.0 PURPOSE

This procedure is to be followed to prolong proper operation of the analytical instrumentation and to minimize and correct defects before they result in serious damage or failure.

2.0 APPLICATION

These procedures apply to the instruments used in the Gas Chromatography/Mass Spectrometry Lab.

3.0 REFERENCES

It is the author's belief that the manuals from the instrument's manufacturer should be the source of the "How-to's" for performing preventive maintenance, routine maintenance, and repairs on all the instruments within the applicable laboratory. In that effort, a library of the manuals supplied with the instrument is located within the specific laboratory location for the reference of the analysts as well as technical support personnel.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 Once a quarter dust off instrument externally and internally or when repairs are made.

5.1.2 Replace septa with new septa as warranted, when the injection port liner is replaced, or after 50 septum punctures.

5.1.3 Replace Injection Port Liner, if applicable, as warranted when column is "clipped".

5.1.4 Cut about a foot off of a capillary column when the peak tailing becomes excessive.

5.1.5 Replace the capillary column when the chromatographic resolution of the column is no longer suitable for the application.

5.1.6 Check zone temperatures via the computer feedback daily or whenever the temperatures have been changed.

5.1.7 Check the operating gases daily for sufficient volume and pressure.

5.1.8 Check the injection port pressure/flow rate and any split flow rates after any connection change on a column or at a minimum of once a month.

5.2 Autosamplers

5.2.1 Replace wash cup septa once a week or when warranted.

5.2.2 Dispose of waste solvent once a week or as warranted.

5.2.3 Perform the internal diagnostics instrument test on the Varian GCs one a month while visually checking the motion of the autosampler for proper action.

5.2.4 Visually check the injection syringe for leaks at the needle connection daily.

5.3 Mass Spectrometers

5.3.1 General

5.3.1.1 Replace/clean all air filters once a quarter.

5.3.1.2 Dust off the instrument externally and internally once a quarter.

5.3.1.3 Check the operation of all cooling fans once a quarter.

5.3.2 Electronics

5.3.2.1 Dust off and reseat all printed circuit boards semi-annually or when any maintenance is performed on that sub-assembly.

5.3.2.2 Reseat all cable connectors whenever any work is performed within the sub-assembly. Reseat all easily accessed cable connectors semi-annually.

5.3.2.3 Readjust RF resonance on the Finnigan MS after each source cleaning or whenever the rear flange is removed from the manifold.

5.3.2.4 Readjust the emission current via the magnet on the Finnigan MS after each source cleaning or whenever the front flange has been removed or the magnet moved.

5.3.2.5 Check the operation of all solenoid valves semi-annually.

5.3.3 Vacuum/Pneumatics System

5.3.3.1 Change the mechanical roughing pump oil semi-annually.

5.3.3.2 Check the oil level in the mechanical roughing pumps and the turbomolecular pumps monthly.

5.3.3.3 Purge the mechanical pumps monthly for about 15 minutes.

5.3.3.4 Replace desiccant in the vent air drier when the blue desiccant has turned to pink in 75% of the drier.

5.3.3.5 Check the condition of the manifold seals each time the flange is removed from the manifold.

5.3.3.6 Check the flexible tubing quarterly or when work is performed on the pumps.

5.3.3.7 Check that the calibration gas valve and introduction system are functional weekly.

5.3.3.8 Leak check pneumatics system if excessive gas consumption is observed.

5.3.3.9 Leak check vacuum system if excessive pressures are observed on the vacuum gauges or if excessive air is present in spectrum.

5.3.4 Data Systems

5.3.4.1 Clean heads on tape units quarterly.

5.3.4.2 Thoroughly clean printers including ribbon transport system semi-annually.

5.3.4.3 Replace printer ribbons as needed, to maintain enough contrast so that no special effort needs to be taken in reproducing copies of the output due to insufficient contrast.

5.3.4.4 Backup your system software at least quarterly, preferably monthly.

5.4 PURGE AND TRAP EQUIPMENT

5.4.1 Leak check all accessible fittings on the LSC-2, ALS, and their connection to the gas chromatograph on a monthly basis.

5.4.2 Methanol flush the LSC-2 and ALS quarterly or when needed as indicated by carryover contamination, plugged or restricted flow, or suspected adsorption problems.

5.4.3 Check flow rates when any line (other than the front panel connections) has been disconnected or connected.

5.5 THERMAL DESORPTION AND CRYOTRAPPING EQUIPMENT

5.5.1 Check temperatures of each zone monthly or more often if stability of the analysis is a problem.

5.5.2 For the Tekmar 4210, use Section 5.4

6.0 DOCUMENTATION

6.1 Each analyst will keep records for the instrument that he is currently stationed at.

6.2 A checklist will be used to record the date and nature of the preventive maintenance performed. (See Figures 1-4).

6.3 Daily/weekly checklists will be stored in the Maintenance Log Book kept with each Mass Spectrometer. Monthly, quarterly, and semi-annually checklists will be stored in a notebook in the possession of the Technical Support Specialist.

6.4 Preventive maintenance documentation will be reviewed monthly by either the Group Leader of the GC/MS Lab or by the Technical Support Specialist.

7.0 DEFINITION

7.1 When a preventive maintenance action is specified to be performed on a daily basis, it is intended to mean each day that the piece of analytical instrumentation is used, and is not required on the days that the instrument is not used.

Weekly and Daily Preventive Maintenance Checklist

Target Date: _____ Instrument: _____

Performed By: _____ Peripherals: _____

Week Beginning: _____

Preventive Maintenance Action	Week	M	T	W	T	F	S	S
Gas Chromatograph								
Check zone temperatures via computer feedback								
Check supply gases for sufficient volume & pressure								
Autosampler								
Check or replace wash cup septa								
Check or dispose of waste solvent								
Visually check injection syringe for leakage								
Mass Spectrometer								
Check calibration gas valve & response level								

Record of routine or conditional preventive maintenance	M	T	W	T	F	S	S
Replacement of GC septa							
Replacement of GC injection port liner							
Clipped GC capillary column							
Replaced GC column							
Cleaned MS source & rods, dipped RF & EC							
Replaced desiccant in vent air drier							
MS manifold seals checked							
MS system leaked checked							
Printer ribbon replaced							
Purge & trap equipment flow rates checked							

Notes & Comments:

Figure 1

Monthly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph ☐ Check the gas pressures and flow rates	_____
Autosampler ☐ Perform Varian instrument test while watching autosampler for proper action	_____
Vacuum Pumps ☐ Check the oil level in the mechanical pumps and in the turbomolecular pumps ☐ Purge the mechanical pumps	_____ _____
Purge & Trap Equipment ☐ Leak check all accessible fittings	_____
Thermal Desorption and Cyrotrapping Equipment ☐ Check temperatures of each zone	_____

Notes & Comments:

Gas Chromatograph/Mass Spectrometry Lab

Figure 2

Quarterly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph	
☐ Dust off instrument internally & Externally	_____
Mass Spectrometers	
☐ Dust off instrument internally & Externally	_____
☐ Replace or clean the air filters	_____
☐ Check the cooling fans	_____
Vacuum Pumps	
☐ Check the integrity of the vacuum tubing	_____
Data Systems	
☐ Clean heads on tape units	_____
☐ Backup the operating software	_____
Purge & Trap Equipment	
☐ Methanol flush LSC & ALS	_____

Notes & Comments:

Gas Chromatograph/Mass Spectrometry Lab

Figure 3

Semi-Annual Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Electronics	
☐ Dust off and reseat all printed circuit boards	_____
☐ Reseat all cable connectors	_____
☐ Check the operation of all solenoid valves	_____
Vacuum Pumps	
☐ Change the mechanical roughing pump oil	_____
Data Systems	
☐ Thoroughly clean printer	_____

Notes & Comments:

Figure 4

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-030-00 Date Initiated: 6/28/89 Page 1 of 7 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the GC Laboratory	2332

Prepared by	Approved by	Date	QA Concurrence	Date
Larry Anderson	A H Caldwell	7/3/89	James M Jones Lauren D Rees Linda P Reeves	7/20/89 7/1/89 7/21/89

1.0 PURPOSE

To outline procedures to be followed for the maintenance and upkeep of the gas chromatographs (GC's), the detectors, and the autosamplers.

2.0 SCOPE

This SOP describes the process of periodic maintenance used on the instruments, detectors, and autosamplers to assure proper performance and quality data.

3.0 REFERENCES

3.1 ITAS-Cincinnati Laboratory Quality Assurance Manual.

3.2 Varian 3300/3400 Gas Chromatograph Operator's Manual, Vol. II.

3.3 Instruction Manual for Model P1-52-02 Photoionization Detector.

3.4 The HALL Book, Tracor Instruments.

4.0 ASSOCIATED SOPS

None.

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 The septum inside the GC injection port is to be changed after each project or at a time determined by the analyst so that the calibration of the instrument is within required specifications.

5.1.2 Each column inside a GC should be constantly watched during an analysis for possible deteriorating performance, such as excessive breakdown, high baseline, or non-resolution of target peaks. If such performance is detected, the column should be pulled from the GC, and the head of the column repacked and the glass wool plug replaced. If in the case of pesticide analysis, separation of A-BHC and B-BHC or endrin ketone and DBC doesn't occur, the column packing should be replaced.

5.1.3 Supply gases are checked every day to make sure that flow to the GCs and detectors are at appropriate levels.

5.1.4 Gas line traps are changed when baseline levels and noise are excessive or when it is determined by the analyst that calibration is outside required specifications.

5.2 Detectors

5.2.1 Electron Capture Detectors

Preventative maintenance on the EC detectors is difficult. However, if an ECD is performing poorly here are several suggestions. First try baking the detector out at about 400°C. This is normally all that is necessary to clean out a dirty detector. If that doesn't work try running hydrogen gas through the lines and into the detector for about 10 minutes. Refer to Varian 3300/3400 Operator's Manual, Vol. II.

5.2.2 Flame Ionization Detectors

FIDs collect a lot of soot from the burning process of the detector, so periodically (every 6 months) the FID is taken apart so the flame tip can be cleaned. The flame tip is first cleaned of soot and dirt with an emery board. A wire brush may help. Then sonicate the flame tip with methylene chloride, hexane, and methanol in that order. It is also a good idea to use the emery paper on the electrodes going to the FID tower. Refer to Varian 3300/3400 Operator's Manual, Vol. II.

When reassembling the detector install a new aluminum seal washer onto the shoulder of the detector base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3300/3400 Operator's Manual, Vol. II.

5.2.3 Photoionization Detector

CAUTION: When disassembling the detector, use disposable rubber gloves or tweezers to avoid contamination of internal components.

The PID is cleaned when the baseline level and the noise are excessive and when it is determined by the analyst that calibration is outside the required specifications. To clean the 10.2 eV UV lamp first turn off the lamp and line power. Allow the detector to cool to ambient temperature before handling. Remove the four lamp housing screws from the rim of the detector assembly and carefully remove lamp housing. Lift the light source from detector. Check the light source window for deposits, films, or discoloration by looking at a reflected image on the window. Then use a clean Kimwipe or other non-linting tissue dampened with water to remove

any water soluble film. Next, add a drop of cleaning compound (supplied by HNU) to the window surface. Use a clean Kimwipe and scour the surface in a circular motion to remove film. Be careful not to scratch the window. Rinse the window with an 80°C solution of mild dish-washing liquid in water and rinse finally with 80°C distilled water. Dry the window in air or with lint free tissue before reassembling the detector. To reassemble the PID, position the disc and lamp window seal in place and place the lamp in position over the lamp window seal making sure the seal is properly positioned to insure against leaks. Carefully place the lamp housing over the lamp and insert and tighten the four screws. Once re-assembled, check the flowrate at the PID exhaust. Refer to PID instruction manual.

5.2.4 Thermionic Specific Detector

This detector is also known as the nitrogen-phosphorus detector. When the TSD exhibits low sensitivity or high background noise cleaning is necessary. Visual inspection may also indicate a need for cleaning since the ceramic bead, collector, or flame tip are all parts that are prone to deposits.

Bead Cleaning: deposits can be removed from the bead by gently scraping the bead surface with a sharp tool or abrasive paper. Support the bead while cleaning to prevent bending the lead and cracking the ceramic coating on the leads.

Flame Tip and Internal Parts Cleaning: handle internal parts with tweezers and place on a clean, uncontaminated surface. Clean deposits from the surface of the collector with water or emery paper. Remove deposits which form on top of the flame tip by scraping. If the flame tip is plugged, clear by inserting wire into flame tip orifice. Flush the cleaned components with methanol and air dry.

When reassembling the detector, install a new seal washer onto the shoulder of the detector base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3300/3400 Operator's Manual, Vol II.

5.2.5 HALL Electrolytic Conductivity Detector

Preventative maintenance for the HALL detector consists of checking to make sure the solvent in the solvent reservoir is at the correct level. The flow from the solvent reservoir must also be checked periodically. A changing flow rate may indicate a leak or clog in the transfer lines. The flow rate should be about 0.5 ml/min. Refer to the HALL book.

5.3 Autosamplers

5.3.1 Dynatech Precision Autosamplers

The septa (2) are replaced once every six months or after it is determined by the analyst that calibration is outside the required specifications of analysis. Close attention is paid to an autosampler when high levels of targets are passed through it to see that no cross-contamination is taking place. Corrective action is taken if such a situation arises. The first injection made by the autosampler is always watched to see if the injection sequence is working properly.

5.3.2 Varian 8000 Autosampler

The septum is replaced once very three months or after it is determined by the analyst that calibration is outside the required specifications of the analysis.

5.4 The Preventative Maintenance Records

5.4.1 Records of preventative maintenance and repairs must be kept in all situations. For this, a logbook has been designed and placed beside each instrument. If an instrument has two detectors that can be used simultaneously, two logbooks will accompany that instrument.

5.4.2 The check list (Figure 1) is filled out by first designating the instrument ID such as V, or A, or K. Next, the week the check list is kept is recorded. The top portion of the list is a quick reference to the availability of the instrument. If for example, an analyst changes the septum and replaces the gas line trap on the instrument the analyst just initials the box pertaining to the action that day.

5.4.3 The bottom portion of the list is filled out when routine maintenance and repairs are made to the instrument. A detailed description of the action performed on the instrument is written down with the analyst's initials and date(s) when the repair took place.

5.4.4 Another quick reference guide used to keep track of when instruments were repaired or maintenanced is on the scheduling board. A tag is placed on the board under the instrument identifier with a small summary of the repair/maintenance on it. This will allow better tracking of when an instrument was maintenanced or when a detector was last cleaned. If more information is needed about the repair, refer to the preventative maintenance check list by the date on the tag.

5.4.5 Preventative maintenance documentation is reviewed monthly by the Group Leader or Team Leader.

WEEKLY PREVENTATIVE MAINTENANCE CHECK LIST WEEK OF _____

INSTR. I.D. _____

ITEM REQUIRED		FREQUENCY	MONDAY	TUESDAY	WEDNESDAY	THURSDAY	FRIDAY	SATURDAY	SUNDAY
CHANGE SEPTUM	FRONT	A,B,I							
	BACK								
CHANGE A/S SEPTUM		F							
ECD(Ni63) WIPE TEST		D							
CHANGE GAS LINE TRAPS		D,E							
REPLACE COLUMN PACKING		D							
CLEAN DETECTOR		D,E							
CHANGE GLASS WOOL PLUG		D,E							

KEY: A. AFTER EACH PROJECT
B. DETERMINED BY ANALYST SO THAT CALIBRATION IS WITHIN REQUIRED SPECIFICATIONS
C. AFTER 3 INJECTIONS
D. ONCE EVERY THREE YEARS - ECD ONLY
E. WHEN BASE LINE LEVEL AND NOISE ARE EXCESSIVE
F. ONCE EVERY THREE MONTHS
I WHICHEVER IS MORE FREQUENT

DATE	INT.	PREVENTATIVE MAINTENANCE / ROUTINE MAINTENANCE

DATE	INT.	REPAIRS MADE TO INSTRUMENT

Figure 1

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-027-00 Date Initiated: 6/26/89 Page 1 of 13 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the Wet Chemistry Laboratory	2332

Prepared by	Approved by	Date	QA Concurrence	Date
Kim Mueller	C.H. Caldwell	6/30/89	James M. Fuller Lauren Drees Linda P. Reeves	7/26/89 7/1/89 7/21/89

1.0 PURPOSE

This SOP describes the maintenance procedures taken on a daily, weekly, or monthly basis to prevent avoidable breakdowns of instruments in the wet chemistry laboratory.

2.0 APPLICATION

This SOP applies to the following instruments:

- Waters Ion Chromatography System
- Technicon TRAACS 800 Auto Analyzer System
- Dohrman/Xertex DC 80 TOC Analyzer
- Dohrman/Xertex DX 10 TOX Analyzer
- Milton Roy Spec 21 UV/Vis spectrophotometer
- Orion Model 720 pH/mv meter
- YSI Model 34 Specific Conductance meter
- Millipore Milli-Q H_2O System

3.0 REFERENCES

3.1 Operator's manual for the Waters model 431 conductivity meter, Model 731 pump, Model 715 Ultra Wisp autosampler.

3.2 Operator's manual for the Technicon TRAACS 800 auto analyzer system.

3.3 Operator's manual for the Dohrman/Xertex TOC analyzer.

3.4 Operator's manual for the Dohrman/Xertex TOX analyzer.

3.5 Operator's manual for the Milton Roy Spec 21 UV/Vis spectrophotometer.

3.6 Operator's manual for the Orion Model 720 pH/mv Meter.

3.7 Operator's manual for the YSI model 34 Specific Conductivity Meter.

3.8 Operator's manual for the Millipore Milli-Q H2O system.

4.0 ASSOCIATED SOPS

None

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Waters Ion Chromatography System

Maintenance	Frequency
Filter and Degas Eluent	With each batch of eluent
Check solvent reservoir filter	With each batch of eluent
Check inlet line for bubbles	Daily, when in use
Check needle wash reservoir	Daily, when in use
Clean or replace guard column	As needed
Clean or replace ion column	As needed
Wipe down outside of instrument	As needed

5.1.2 TRAACS Auto Analyzer System

Maintenance	Frequency
Check air pressure (22 psi +/- 2)	Daily, when in use
Remove tubing from air valves	Daily, when in use
Check base and gain settings	Weekly, when in use

(continued)

Maintenance	Frequency
Change pump tubing	Monthly
Replace pump platens	Every 6 months
Replace colorimeter lamp	Yearly
Replenish reagents	As needed
Wipe down outside of instrument	As needed

5.1.3 Dohrman/Xertex DC 80 TOC Analyzer

Maintenance	Frequency
Replace septa	As needed
Replace glass wool in boat	Daily, when in use
Replenish reaction vessel reagent	Daily, when in use
Repack tin and copper scrubbers	As needed
Replace pump tubing	As needed
Repack lithium hydroxide scrubber	As needed
Repack cupric oxide furnace tube	As needed
Wipe down outside of instrument	As needed

5.1.4 Dohrman/Xertex DX 10 TOX Analyzer

Maintenance	Frequency
Check inlet and outlet tubes for residue buildup	Daily, when in use
Clean or replace inlet and outlet tubes	As needed
Check all electrodes for residue buildup	Daily, when in use
Clean or replace cell electrodes	As needed
Replenish electrolyte solution	As needed
Replace glass columns and "O" rings	As needed
Wipe down outside of instrument	As needed

5.1.5 Milton Roy Spec 21 UV/Vis Spectrophotometer

Maintenance	Frequency
Check instrument response with commercially purchased solutions	Monthly
Check wavelength with didymium filter	Monthly
Wipe down outside of instrument	As needed

5.1.6 Orion Model 720 pH/mv Meter

Maintenance	Frequency
Clean electrode	As needed
Wipe down outside of instrument	As needed

5.1.7 YSI Model 34 Specific Conductivity Meter

Maintenance	Frequency
Clean conductivity cell	Quarterly
Replatinize cell	As needed

5.1.8 Milli-Q H_2O System

Maintenance	Frequency
Change deionizing tanks	As needed
Change Milli-Q cartridges	As needed
Conductivity check	Daily

5.2 Documentation of Maintenance

5.2.1 Each time a maintenance check or procedure is performed it is recorded on the appropriate maintenance log sheet (Figures 1-8) and kept in books near each instrument.

5.2.2 When a routine check or procedure is performed, the analyst dates and initials the appropriate line on the log sheet. If a replacement is made, it is noted in the space provided for additional information.

5.2.3 When a non-routine maintenance procedure is performed, a description is entered in the space provided for additional information as well as the date and the name of the person performing the procedure.

5.2.4 All preventive maintenance documentation is reviewed by the lab supervisor monthly.

PREVENTIVE / ROUTINE MAINTENANCE
WATERS ION CHROMATOGRAPHY SYSTEM

Daily Basis

Check Inlet Line for Bubbles & Needle Wash Reservoir

Date	Initials	Date	Initials

Whenever New Eluent is Made

Eluent Filtered & Degassed, & Solvent Reservoir checked

Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Clean guard column		
Replace guard column		
Clean ion column		
Replace ion column		

Checked By:_____ Date:_____

Figure 1

PREVENTIVE / ROUTINE MAINTENANCE
TRAACS 800 AutoAnalyzer System

Daily Basis

Check Air Pressure & Remove Tubing from Air Valves

Date	Initials	Date	Initials

Weekly Basis

Check Base & Gain Settings

Date	Initials	Date	Initials

Monthly Basis

Replace Pump Tubings

Date	Initials

6 Month Basis

Replace Pump Platens

Date	Initials

Yearly Basis

	Date	Initials
Replace Colorimeter Lamp		

Checked By:_____ Date:_____

Figure 2

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DC 80 TOC ANALYZER

Daily Basis

Replace Glass Wool

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Replenish Persulfate Sol'n

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Repack Tin & Copper Scrubbers		
Replace pump tubing		
Repack Lithium Hydroxide Scrubber		
Repack Cupric Oxide tube		

Checked By:_____ Date:_____

Figure 3

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DX 20 TOX ANALYZER

Daily Basis

Check Inlet & Outlet Tubes For Residue Buildup

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Check Cell Electrodes For Residue Buildup & Replenish Electrolyte

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Clean Inlet & Outlet Tubes		
Replace Inlet & Outlet Tubes		
Clean Cell Electrodes		
Replace Cell Electrodes		

Checked By:_____ Date:_____

Figure 4

PREVENTIVE / ROUTINE MAINTENANCE
MILTON ROY SPECTRONIC 21 UV/VIS SPECTROPHOTOMETER.

Monthly Basis

Check Response with Commercially Prepared Solutions

Date	Initials	Date	Initials

Monthly Basis

Check Wavelength with Didymium filter

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 5

PREVENTIVE / ROUTINE MAINTENANCE
pH METER & ELECTRODE

Monthly Basis

Clean Electrode & Meter

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 6

PREVENTIVE / ROUTINE MAINTENANCE
CONDUCTIVITY METER

Quarterly Basis

Clean Cell & Meter

Date	Initials	Date	Initials
_____	_____	_____	_____
_____	_____	_____	_____

As Needed Basis

Re-Platinize Cell

Date	Initials	Date	Initials
_____	_____	_____	_____
_____	_____	_____	_____

Additional Maintainence Documentation

_____
_____
_____
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_____

Checked By:_____ Date:_____

Figure 7

PREVENTIVE / ROUTINE MAINTENANCE
D.I. H2O QUALITY SYSTEM

Change D.I. Tanks

As Needed Basis

Date	Initials	Date	Initials

Change Milli-Q Cartridges

As Needed Basis

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 8

APPENDIX C

CLP PERFORMANCE EVALUATION RESULTS AND AUDIT FOR IT ANALYTICAL SERVICES CINCINNATI, OHIO

ONTRACT
VIDENCE
UDIT
EAM

October 3, 1989

Mr. Howard Fribush
Project Officer (WH-548A)
USEPA Headquarters
Office of Solid Waste and
Emergency Response
Analytical Operations Branch
401 M Street, S.W.
Washington, DC 20460

RE: Transmittal of CEAT Laboratory Evidence Audit Report for PEI Associates, Inc.

Dear Howard:

Enclosed is a copy of the Contract Evidence Audit Team (CEAT-TechLaw) laboratory evidence audit report for the audit conducted at PEI Associates, Inc. on September 13, 1989.

Procedures and documentation related to sample receiving, sample storage, sample identification, sample security, sample tracking, and case file organization and assembly were reviewed for conformance to Evidence Audit Requirements. Non-conformances to Evidence Audit Requirements are expressed as findings in the attached report. Recommendations for corrective action are discussed in the "Executive Summary" of the report.

If you have any questions, please contact the Deputy Project Officer, Paula Smith, at (303) 236-5122, FTS 776-5122.

Sincerely,

Jeffrey C. Worthington
Contract Evidence Audit Team

Concurrence:

Paula Smith
National Enforcement
Investigations Center

srw

Enclosure

cc: Pat Churilla, USEPA Region V DPO
Karen M. Drees, PEI Associates, Inc.

IF: 111-001

LABORATORY EVIDENCE AUDIT REPORT

PEI ASSOCIATES, INC.

September 13, 1989

PEI Associates, Inc.
11499 Chester Road
Cincinnati, OH 45246
(513) 782-4700

Lauren M. Drees	- QC Coordinator[1,3]
H. William Jess	- Laboratory Supervisor[1,2,3]
Lawrence A. Elfers	- Vice President[1,3]
Craig Caldwell	- Laboratory Manager[1,3]
Dawn Webber	- Document Control Officer[2]
Lauri Rotella	- Sample Custodian[2]
Craig Stoll	- Extraction Group Leader[2]
Craig Crume	- GC Group Leader[2]

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(312) 353-2313

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A. Bill Kieger - Associate Consultant
Elizabeth Murray - Staff Associate

[1]present at pre-audit briefing
[2]contacted during audit
[3]present at post-audit debriefing

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA contract #68-01-7369.

INTRODUCTION

An audit of laboratory operations pertaining to laboratory security, sample chain-of-custody, and document control procedures for EPA organics contracts 68-01-7474 (IFB WA 87-K238), 68-01-7467 (IFB WA 87-K236), and 68-D9-0037 (IFB W802036D1) was conducted at PEI Associates, Inc. (PEI) in Cincinnati, Ohio, on September 13, 1989. The audit was conducted by NEIC's Contract Evidence Audit Team (CEAT-TechLaw). Procedures and documentation related to sample receiving, sample storage, sample security, sample tracking, and case file organization and assembly were reviewed for conformance to Evidence Audit Requirements. The results of this audit are discussed in this evidence audit report.

EXECUTIVE SUMMARY

This was the fourteenth routine organics audit of PEI conducted by USEPA representatives in support of the Contract Laboratory Program (CLP). The previous audit was conducted on August 10, 1988, and resulted in nine recommendations from the CEAT. Eight of these recommendations have been addressed and corrected. The one recommendation from the previous audit still requiring corrective action is:

1. Laboratory documents should include identification of all activities performed.

Findings

The following five findings (non-conformances to Evidence Audit Requirements) were identified during the present audit and are discussed in this report:

1. The Sample Control Record, GPC Logbook, and Instrument Run Log did not contain the name of the laboratory.

2. The GPC Logbook, Laboratory Notebook documenting base/neutral/acid (BNA) and pesticide preparation, and the Instrument Run Log did not include identification of the activities performed.

3. The reviewer's signature was not clearly identified in the GPC Logbook.

4. Corrections to supporting documents were not consistently made by drawing a single line through the error and entering the correct information. Corrections to supporting documents were not consistently dated and initialed.

5. The Re-Extract/Clean-Up Request forms were not included in the case files prior to submission to EPA/NEIC.

Recommendations

As a result of the findings, the following recommendations were made during the debriefing with laboratory personnel at the conclusion of the audit on September 13, 1989:

1. The Sample Control Record, GPC Logbook, and Instrument Run Log should contain the name of the laboratory.

2. The GPC Logbook, the BNA/Pesticide Preparation Logbook, and the Instrument Run Log should clearly identify the activities being performed. The reviewer's signature should be clearly identified in the GPC Logbook.

3. Corrections to supporting documents should be made by drawing a single line through the error and entering the correct information. Corrections to supporting documents should be dated and initialed.

4. The Re-Extract/Clean-Up Request forms should be included in the EPA case files prior to submission to EPA/NEIC.

Routine evidence audits will be conducted during the contract period of performance. Corrective action on the above items will be reviewed during the next on-site audit. Periodic audits will be conducted to review continued conformance to Evidence Audit Requirements.

The audit was concluded on September 13, 1989. The audit participants are listed on the cover page of this report.

PROCEDURAL AUDIT

The procedural audit consisted of review and examination of actual and written standard operating procedures (SOPs) and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample tracking (from receipt to completion of analysis), and case file organization and assembly.

Sample Receiving

EPA sample shipments are received at the Sample Control Room on the ground floor of the laboratory facility. L. Rotella, the designated sample custodian, inspects the containers and custody seals before the samples are unpacked. The containers are then unpacked, information recorded on accompanying documentation is verified for agreement, and sample receipt information is entered into a computer.

The Sample Management Office (SMO) is contacted to resolve problems with the samples or shipping information, and the resolution is recorded in the custodian's telephone log.

Written SOPs for sample receiving have been developed and implemented and are documented in PEI's SOPS titled Sample Receipt, Log-In, and Storage and CLP Sample Receipt. The auditors read these SOPs, and they accurately describe the procedures in use for sample receipt.

Sample Storage

Semi-volatile samples are stored in a walk-in cooler located in the hallway outside the Sample Control Room. Sample extracts and volatile organic (VOA) samples are stored in upright refrigerators (refrigerators #2 and #3, respectively) located in a room adjacent to the Sample Control Room. Extracts are temporarily stored on a bench in the Preparation Laboratory before being transferred to permanent storage.

Samples are identified with the PEI number and Sample Delivery Group (SDG) number. Extracted samples are identified with the SMO number, PEI number, case number, SDG number, and fraction type. During sample preparation, sample identity is maintained by recording the SMO number, PEI number, case number, and fraction type on all intermediate vessels.

Sample and laboratory security is maintained by keeping all doors to the laboratory areas locked. Authorized personnel gain access to these areas through the use of keys. All visitors are escorted throughout the laboratory areas.

Written SOPs for sample storage, security, and identification have been developed and implemented and are documented in PEI's SOPs titled Sample Receipt, Log-In, and Storage and Chain of Custody and Sample Security. The auditors read these SOPs, and they accurately describe the procedures in use for sample storage, security, and identification.

Sample Tracking

The following is a summary of the laboratory documents that are used to track samples (from receipt to completion of analysis) and the activities recorded on these documents:

	Document Title	Activity(s) Recorded
1.	Work Order	Sample Receipt/Tracking
2.	Sample Control Record	Sample Tracking
3.	GC Screen Logbook	Gas Chromatograph (GC)/Pesticide Screen
4.	GPC Logbook	Gel Permeation Column (GPC) Clean-Up
5.	Laboratory Notebook	BNA and Pesticide Preparation
6.	Extraction Summary	Percent Moisture and pH Determination
7.	Re-Extract/Clean-Up Request Form	BNA and Pesticide Preparation
8.	Instrument Run Logs	GC Analysis
9.	Run Logbooks	BNA and VOA Analysis by Gas Chromatograph/ Mass Spectrometry (GC/MS)
10.	Sample Dilution Record	Sample Dilution

The documents appeared to provide a complete record of all activities observed by the auditors. The following non-conformances to Evidence Audit Requirements were noted by the auditors:

1. The name of the laboratory did not appear on Items 2, 4, and 8 listed above.

2. Item 4, listed above, did not clearly identify the reviewer's signature.

3. Items 4, 5, and 6, listed above, did not include identification of the activities performed.

4. Error corrections were not consistently signed and dated on Items 8 and 9 listed above.

5. Item 7, listed above, was not included in the case files prior to submission to EPA/NEIC.

Written SOPs for sample tracking have been developed and implemented and are documented in PEI's SOPs titled CLP Sample Tracking and Chain of Custody and Sample Security. The auditors read these SOPs, and they accurately describe the procedures in use for sample tracking.

Case File Organization and Assembly

Case file documents are arranged by EPA case number and are stored in a locked file room adjacent to the GC/MS Laboratory. D. Webber is responsible for assembling organic case files. She numbers and inventories the documents in each case file and checks for completeness. She then enters all inventory information into a computer and generates an inventory form, which is signed and dated and included in each case file.

According to D. Webber, PEI has not received any EPA-designated confidential documents.

Written SOPs for case file organization and assembly have been developed and implemented and are documented in PEI's SOPs titled CLP Case File Assembly, Storage, and Purging. The auditors read these SOPs, and they accurately describe the procedures in use for case file organization and assembly.

EVIDENCE AUDIT

The evidence audit consisted of review and examination of case file documentation. Case files contain the following types of documents:

1. Document File Inventory
2. Airbill
3. Chain of Custody Record
4. Traffic Report
5. Extraction Summary
6. Sample Control Record
7. Sample Tags
8. BNA Instrument Logs

9. GC Instrument Logs
10. VOA Instrument Logs
11. Extraction Notebook and Logbook pages
12. SDG Cover Sheet
13. Contract Compliance Screening Summary
14. Case Narrative
15. Data Package
16. Work Order

The case files examined during the audit were 12015, 12011, and 12469.

Documents in the case files are organized and developed according to Evidence Audit Requirements except for the following non-conformances:

1. Items 6, 8, and 9, listed above, did not contain the name of the laboratory.

2. Items 8, 9, and 10, listed above, did not include identification of the activities performed.

3. Corrections to Items 9, 10, and 16, listed above, were not consistently made by drawing a single line through the error and dating and initialing the correction.

SUMMARY

At the conclusion of the audit on September 13, 1989, a debriefing was held by the audit team with laboratory personnel. During this debriefing, the evidence auditors identified their findings and made recommendations for corrective action. The findings and recommendations are presented in the "Executive Summary" of this report.

INTERNATIONAL TECHNOLOGY CORPORATION

MEMORANDUM

To: Laboratory Supervisors

Date: 8/17/89

Subject: CLP Audit

From: Lauren LMD

Representatives for the Contract Laboratory Program will be auditing our laboratory on Wednesday, September 13. After reviewing the findings from the previous audit, I have determined that some items still need to be addressed. These items are summarized below:

1) The SOP for sample tracking needs revision. This SOP descibes the documentation generated with the samples from the time of receipt to the time of purging. Lauri and I will be responsible for this SOP.

2) An SOP specific to CLP sample receipt must be prepared. It must include where resolutions to problems or discrepancies are documented. Lauri and I will also be responsible for this SOP.

3) The SOP for case file organization and assembly needs to be revised. Procedures to ensure that case files are purged 180 days after analyses must be included. Craig Caldwell and Lauri are responsible for this SOP.

4) Standards traceability SOPs must be prepared in the extractions, GC/MS, and GC lab areas. Craig Stoll, Dave Neal and Craig Crume, respectively, are responsible. I have distributed a draft EPA procedure sent to me after the last audit; please see me if you need another copy to use for guidance.

Some other things that should be reviewed in preparing for the audit include method and holding blank documentation, run logs, preventive maintenance logs, standards logs, solvent checking and alumina equivalency documentation, hood flow monitoring logs, balance logs, and refrigerator/oven logs.

Attached is a copy of the report from the last audit (August, 1988). Please review and take action where possible. Also attached are the results of a data review for Case 10876; problems will be discussed during the audit. Please be prepared identify possible sources of error.

If you have any questions or problems, please ask.

CONTRACT EVIDENCE AUDIT TEAM
LABORATORY AUDIT ATTENDEES LIST
(please print)

LABORATORY NAME PEI Associates, Inc. DATE 9/13/89

Name	Affiliation	Position	Telephone
A. Bill Krieger	TechLaw/NEIC	Associate Consultant	(303) 233-1248
Elizabeth Murray	TechLaw/NEIC	Staff Associate	(303) 233-1248
Larry ELFERS	PEI	Lab Director	513-782-4600
Craig Caldwell	PEI/ITAS	Lab Manager	"
H. W. JESS	PEI	LAB Supervisor	
Lauren Drees	PEI	QC Coordinator	513-782-4600
Jim Pels	USEPA-Reg II	Chemist	(212) 353-2720

LF Debriefing 9/13/89

Recommendations (TechLaw)

1. All bench sheets need laboratory name.
2. " " " " to identify activity being performed. (inc. reviewed by)
3. Error corrections.
4. All EPA case related documents must be included in case file. (Re-extraction form).

Findings (Region V)

1. Change SOP containing 2-butanone internal standard

Seymour

Replace canister SOP to current one. Add surrogates to SOP. Control charts. Use Scott gas mixture to verify calibration.

ORGANIC PERFORMANCE EVALUATION SAMPLE
INDIVIDUAL LABORATORY SUMMARY REPORT
FOR QB 3 FY 90

LABORATORY: PEI Associates (OH)
PERFORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 14 Same = 0 Below = 31

% SCORE: 92.1
REPORT DATE: 07/03/90
MATRIX: WATER

COMPOUND	CONFIDENCE INTERVALS WARNING LOWER	WARNING UPPER	ACTION LOWER	ACTION UPPER	LABORATORY DATA CONC	Q	#LABS MIS-QNT	PROGRAM DATA #LABS NOT-ID	#LABS ID-CPD	TOTAL #LABS
TCL VOLATILE										
VINYL CHLORIDE	22	36	20	38	27		6	0	46	46
CHLOROETHANE	23	35	21	37	26		2	0	46	46
ACETONE	52	133	40	145	130		3	1	45	46
CARBON DISULFIDE	35	55	32	58	48		6	0	46	46
1,2-DICHLOROETHENE (TOTAL)	73	102	69	106	95		6	0	46	46
BROMODICHLOROMETHANE	47	58	45	60	54		4	0	46	46
DIBROMOCHLOROMETHANE	61	78	58	80	71		8	0	46	46
BROMOFORM	43	58	40	60	48		1	0	46	46
1,1,2,2-TETRACHLOROETHANE	16	22	15	23	17		6	0	46	46
STYRENE	67	96	62	100	76		4	0	46	46
TCL SEMIVOLATILE										
BIS(2-CHLOROETHYL)ETHER	29	50	26	53	35		1	1	45	46
2-CHLOROPHENOL	80	128	73	154	77	S	0	0	46	46
1,2-DICHLOROBENZENE	46	84	41	104	63		2	0	46	46
BIS(2-CHLOROISOPROPYL)ETHER	38	67	34	71	42		1	0	46	46
4-METHYLPHENOL	43	64	40	76	38	X	2	0	46	46
N-NITROSO-DI-N-PROPYLAMINE	49	78	44	82	51		2	0	46	46
2,4-DIMETHYLPHENOL	45	74	40	90	44	S	1	0	46	46
BIS(2-CHLOROETHOXY)METHANE	30	50	28	52	31		3	0	46	46
2-METHYLNAPHTHALENE	30	56	26	60	40		3	0	46	46
HEXACHLOROCYCLOPENTADIENE	11	54	10	78	46		0	7	39	46
2,4,6-TRICHLOROPHENOL	50	74	47	86	49	S	0	0	46	46
2,6-DINITROTOLUENE	31	46	29	54	36		2	0	46	46
ACENAPHTHENE	44	67	41	70	49		4	0	46	46
2,4-DINITROPHENOL	50	95	50	102	54		0	2	44	46
DIBENZOFURAN	65	97	60	101	75		4	0	46	46
DIETHYLPHTHALATE	27	103	15	115	21	S	10	3	43	46
FLUORENE	55	80	51	83	60		3	0	46	46
4-NITROANILINE	56	115	50	123	49	S	3	1	45	46
PHENANTHRENE	68	102	63	107	70		3	0	46	46
FLUORANTHENE	68	105	62	111	71		1	0	46	46
BUTYL BENZYL PHTHALATE	22	83	13	92	17	S	9	3	43	46
3,3'-DICHLOROBENZIDINE	47	125	35	136	53		6	0	46	46
BIS(2-ETHYLHEXYL)PHTHALATE	52	90	46	96	59		4	0	46	46
DI-N-OCTYL PHTHALATE	52	88	47	94	53		3	0	46	43
INDENO(1,2,3-CD)PYRENE	63	99	58	104	70		7	0	46	46
DIBENZ(A,H)ANTHRACENE	64	102	58	108	73		6	0	46	46
BENZO(G,H,I)PERYLENE	65	101	60	106	76		5	0	46	46
TCL PESTICIDES										
ALPHA-BHC	0.38	0.72	0.33	0.77	0.46		5	0	46	46
BETA-BHC	0.3	0.56	0.26	0.6	0.38		4	0	46	46
DELTA-BHC	0.25	0.5	0.21	0.54	0.37		3	0	46	46
GAMMA-BHC (LINDANE)	0.34	0.64	0.29	0.69	0.38		4	0	46	46
ENDOSULFAN I -	0.34	0.59	0.3	0.62	0.61	S	1	1	45	46

ORGANIC PERFORMANCE EVALUATION SAMPLE
INDIVIDUAL LABORATORY SUMMARY REPORT
FOR QB 3 FY 90

:BORATORY: PEI Associates (OH)
:FORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 14 Same = 0 Below = 31

% SCORE: 92.1
REPORT DATE: 07/03/90
MATRIX: WATER

:POUND	CONFIDENCE INTERVALS WARNING LOWER	WARNING UPPER	ACTION LOWER	ACTION UPPER	LABORATORY DATA CONC	Q	#LABS MIS-QNT	PROGRAM #LABS NOT-ID	DATA #LABS ID-CPD	TOTAL #LABS
:CLOR-1260	3.3	5.2	3	5.5	4.2		5	0	46	46
:-TCL VOLATILE										
PANE,1,2-DIBROMO-3-CHLORO-					9			14	32	46
HANE,IODO-					90			3	43	46
:-TCL SEMIVOLATILE										
:ZOPHENONE					0	&		3	43	46
:ZILATE,CHLORO-					0			24	22	46
:ENE,BENZO(E)-					0			40	6	46
:IDINE					30			35	11	46
NONE,1,4-NAPHTHO-					0			37	9	46
SEMIVOLATILE (Contaminants)										
N-BUTYLPHTHALATE					2			43	3	46
'ZO(A)PYRENE					81			20	26	46
'-TCL SEMIVOLATILE (Contaminants)										
-NAPTHALENEDIONE,2-HYDR					10			24	22	46
,4-TRIOXOLANE,3,3,5-TRIP					50	C		45	1	46

F TCL COMPOUNDS NOT-IDENTIFIED: 0
F TCL COMPOUNDS MIS-QUANTIFIED: 1
F TCL CONTAMINANTS: 0

F NON-TCL COMPOUNDS NOT-IDENTIFIED: 1
F NON-TCL CONTAMINANTS: 1

APPENDIX D

COMMENTS ON VOLUME

This appendix was attached to the front of the August 1990 Draft. The comments relevant to this volume have been incorporated into this issuance.

DEPARTMENT OF THE AIR FORCE

HEADQUARTERS 2750TH AIR BASE WING (AFLC)
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-5000

31 AUG 1990

Y TO
OF EM

ECT Completion of Work Plans, Off-Site Groundwater Investigation, Wright-Patterson Air Force Base

TO See Distribution

1. Attachments 1, 2, and 3 provide our comments, Ohio EPA comments (dated 20 Aug 90) and additional technical information respectively required for the subject work plans. The addition of this letter and the attachments to the front of Volumes 2-4 and Volume 3 Appendix A shall be considered sufficient to complete these work plans. The terms and conditions, as specified in the Ohio EPA letter dated 20 Aug 90, will be followed during the Field Investigation.

2. Should you have any questions or require additional copies of this letter, please contact Mr Gary W. Selby, (513) 257-2201.

FOR THE COMMANDER

John A. Nepute

JOHN A. NEPUTE, P.E., Acting Deputy Director
Office of Environmental Management

3 Atch
1. EMR Comments, 20 Aug 90
2. OEPA Comments, 20 Aug 90
3. Additional Technical Information

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Mr Henry Hunter
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Fairborn OH 45324

Mr William Thompson
IT Corporation
11499 Chester Road
Cincinnati OH 45246

HQ AFLC/DEVR

COMMENTS ON VOLUME 3, SAMPLING AND ANALYSIS PLAN

1. List of Acronyms: "Assistant" is misspelled in the first line. Also, the following acronyms should be added:

EMR	Environmental Management, Restoration Branch
FI	Field Investigation
FFS	Focused Feasibility Study
mg	milligram
ml	milliliter
L	Liter
TDS	Total Dissolved Solids

2. Section 1.0: "CERCLA" should be noted as being the Comprehensive Environmental Response, Compensation, and Liability Act.

3.Section 3.1: "management operations" should be capitalized in fourth line.

4. Section 3.1.1, first paragraph: "base" should be capitalized in the first line. "EM" should be "OEM" in the third line.

5. Page 3-8, fourth bullet: "RI report" should be "Field Investigation Report".

6. Table 4-1: "water" should be deleted from the Parameter column.

7. Table 5-1: "HD-12S" should be added to Cluster CW6.

8. Page 5-12, first bullet: "instruments" is misspelled. "or 10 ppm" should be deleted.

9. Page 5-12, second bullet: "reproducible" should be added after "If".

10. Page 5-12, first full paragraph: "the boreholes will be abandoned" should be added after "necessary" in the first sentence.

11. Page 5-12, last line: "coarse" should be deleted.

12. Page 5-15, first full bullet: "EPA" should be "OEPA".

13. Section 5.4.6: "but no more than 2 days" should be added to the second sentence.

14. Page 5-18, second paragraph: "in accordance with Federal, State and local regulations" should be added to the second sentence.

15. Page 6-1: "signature(s) of the person(s) relinquishing and accepting sample custody, and other pertinent information." should be added to the last sentence on this page to make it complete.

16. Section 8.4.3.3.3: "each" is misspelled in the second line.

17. Section 9.1, first paragraph: "TPM" should be "TPH" in the third sentence.

18. Page 10-4, second full paragraph: "the" should be added after "of" in the first sentence.

19. Page R-1: The Engineering-Science RI/FS Work Plans (Volumes 1 and 2, Volume 2 Appendix C) should be added to this list.

OhioEPA
ate of Ohio Environmental Protection Agency
outhwest District Office
South Main Street
yton, Ohio 45402-2086
13) 285-6357
X (513) 285-6249

Hand-delivered to WPAFB
Aug 20, 1990

Richard F. Celeste
Governor

August 20, 1990 Re: Workplan for the Investigation of Ground Water Contamination at WPAFB

Scott Mallette, Chief
Environmental Restoration Branch
2750 ABW/EM (AFLC)
Wright-Patterson Air Force Base, Ohio 45433

Dear Mr. Mallette:

With exceptions noted, the following comments on the "Workplan, Phase I Task 4 Field Investigation" were discussed with Gary Selby, Denny Reed, and Bill Thompson at the August 13, 1990 progress meeting. It is Ohio EPA's understanding that the Air Force had no objections to the comments discussed at that meeting and that the comments will be incorporated into the work to be performed during the Phase I Task 4 investigation. It is also Ohio EPA's understanding that the drilling subcontractor was notified to mobilize so as to be able to start work on this project by September 5, 1990. Ohio EPA hereby concurs with the "Workplan, Phase I Task 4 Field Investigation" with the following four conditions:

1. All of Ohio EPA's comments appearing below will be incorporated into the work to be performed during this investigation.

2. In the interest of time, and with the intent of avoiding any delays in the start date for this investigation, the Workplans for the project will not be revised by the Air Force or resubmitted to Ohio EPA. In lieu of revision, this comment letter will be copied by the Air Force and bound into the front of each of the separate volumes of the Phase I Task 4 Field Investigation Workplan so as to become part of that Workplan.

3. Ohio EPA's August 2, 1990 correspondence containing comments on the Field SOPs for the RI/FS Workplan will be incorporated into the Phase I Task 4 field work. The RI/FS Field SOPs in combination with the procedures outlined in the RI/FS Workplan will be followed during the Phase I Task 4 field work except where modified by Ohio EPA approved project-specific amendments.

4. The Air Force will provide Ohio EPA with written confirmation that the conditions outlined above are understood and will be met during the Phase I Task 4 Field Investigation. This written confirmation is to be provided to Ohio EPA no later than Tuesday, August 28, 1990.

Ohio EPA Comments - Volume 2, Workplan for Phase I Task 4 Field Investigation - (August 6, 1990)

1. Page iii, Table 4-3: Correct page # for Table 4-3 is 4-17.

2. Page 2-4, third line from bottom: Typo - "product" should be "production"

3. Page 3-1, 3.1, second paragraph: Typo - "alternative" should be "alternatives".

4. Page 3-3, 3.2.1: "Dependant" should be spelled "dependent".

5. Page 3-3, 3.2.1: The confining layer would need to be under the area to be controlled, not over.

6. Page 3-6, 3.2.3, last sentence of first paragraph: Delete the word "or" from this sentence. Contaminants may have already migrated beyond the WPAFB boundary to a point where they are affecting Dayton's well field. This may indeed require treatment at the well field. This does not mean that an interception system designed to prevent further off-base contaminant migration will not be necessary at the base boundary, although this is what seems to be implied by the current wording of this sentence. It must be clearly understood that treatment at Dayton's well field is not an acceptable substitute for contaminant control at the base boundary.

7. Page 3-6, 3.2.3, last sentence on page: "Table 3-5" should read "Table 3-2".

8. Page 4-2, 4.1.1, second sentence: Delete this sentence. The referenced criteria are not used in the screening of remedial alternatives.

9. Page 4-3, third paragraph: Specify that the existing guidance being referred to is U.S. EPA's "Interim Guidance for Preparing Quality Assurance Project Plans", (QAMS-005/80).

10. Page 4-5, 4.2, second paragraph: Reword to read: "Before the effectiveness of control or removal programs is evaluated, a numerical model will be developed. Additional

site-specific data must be collected to calibrate the model. These data will be generated through a field investigation performed as a part of this overall ground water study."

11. Page 4-5 and 4-6, 4.2.1: Indicate in this section that observation wells will be installed during future phases of the project to verify the model-predicted capture zone scenarios.

12. Page 4-6, second paragraph, first sentence: Delete the phrase "...to control contaminant migration..." from this sentence.

13. Page 4-6, third paragraph, second sentence: Delete the first word "As" and start the sentence "The existing data base...".

14. Page 4-9, 4.2.2: Include the pumping rates and schedules for WPAFB's Area B and Area B East well fields as data needs which will need to be factored into the model.

15. Page 4-9, 4.2.3, last sentence: Typo - Change "be" to "the".

16. Page 4-10, first full sentence: Delete the word "that" so the sentence reads ""While several production wells located upgradient from Base boundaries in Area B yield water with low levels of...".

17. Page 4-13, top of page: Replace the phrase "back-end project requirements" with the phrase "project scheduling constraints" and explain how those constraints affect the number of wells installed. Note that Ohio EPA expects to see 22 monitoring wells installed during the Phase I investigation (see Table 4-2 on page 4-15).

18. Page 4-13, 4.3.3: Identify the purpose behind sampling the six wells for gross water quality. Indicate that Ohio EPA concurrence with the selection of these six sampling points will be obtained prior to the samples being collected.

19. Page 4-13, 4.3.3: Indicate that monitoring wells along the Springfield Street boundary will be sampled at a time when the adjacent WPAFB water supply production wells are not operating. A waiting period after shut down should be established which is sufficient to allow flow conditions in the area to return to prepumping conditions prior to sampling the monitoring wells.

20. Page 4-17, Table 4-3: Include Carbon tetrachloride and Bromomethane in the TCL Volatile Organic Compounds.

21. Page 6-1, 6.0: Replace the phrase "back-end deadlines" with the phrase "project scheduling constraints".

22. Page 7-4, Figure 7-2: The names in this table are not current. Please update the table.

Ohio EPA Comments - Volume 3, Sampling and Analysis Plan (SAP) Phase I Task 4 - (August 8, 1990)

1. Comments provided above that apply to sections of the SAP which are identical to sections in Volume 2 are to be incorporated into the SAP. These comments have not been repeated below.

2. Page 5-10, 5.4.1: The drillers and site geologist must be sensitive to changes in lithology during the drilling of the deep pilot hole. Although a lithologic sample will be collected every five feet, the bore hole will likely be bailed more frequently. The cutting should be visually examined for gross changes in lithology each time the bailer is withdrawn from the bore hole, and a lithologic sample needs to be collected each time a change in lithology is detected in addition to those collected every five feet.

3. Page 5-10, 5.4.1: Driving the casing may prove difficult through parts of the formation, particularly the boulder zone. Refer to the attached table of recommended casing standards excerpted from Ohio EPA's Water Well Standards (OAC 3745-9-06) for assistance in determining which well casing is appropriate for the project.

4. Page 5-12, first and second bullets: Drill cuttings are to be containerized if any reproducible readings above background are obtained with the field screening instruments. Delete the reference to 10 ppm which appears in these two bullets and revise accordingly. Any soils which are determined to be contaminated are at least solid waste and may be hazardous waste. Procedures to be followed in testing and disposing of project generated wastes are those identified in the RI/FS Field SOPs and the RI/FS Workplan.

5. Page 5-12, first paragraph: The first two sentences need to be combined using a comma after the OAC reference.

6. Page 5-12, last sentence carrying over to 5-13: The sand pack is to consist of coarse silica sand.

7. Page 5-13, 5.4.4: Indicate that if the turbidity requirements for developed wells cannot be met after eight hours of development, samples will be collected and analyzed for silt, clay and total organic carbon per the procedures described in FP5-4 and the RI/FS Workplan to determine the cause of the turbidity failures. It is understood that well development will not continue more than eight hours for purposes of this project regardless of the turbidity measurements obtained at the end of the development period. However, it is necessary to determine the cause of the turbidity failures in order to properly interpret contaminant data obtained from the well. Please also note that visual inspection is not the criteria by which turbidity is determined. A nephelometer is to be used.

8. Page 5-14, 5.4.5, fourth bullet: Indicate that a clear bailer will be used to determine the presence or absence of floating free product.

9. Page 5-15, first bullet: Change EPA to Ohio EPA in the last sentence of this bullet.

10. Page 5-16, 5.4.6: If at all possible, water levels should be obtained from all wells on the same day. If that is not possible, the water levels should be collected over a period not to exceed two consecutive days.

11. Page 5-18, second paragraph, last sentence: Indicate that disposal of decontamination fluids will conform with federal, state, and local laws. Ohio EPA expects that these fluids will be handled in a manner identical to the procedures for handling project generated wastes as worked out with the Air Force during the RI/FS Workplan comment resolution meetings. All wastes generated during the Off-Site investigation are to be handled following these procedures.

12. Page 5-23, second paragraph: This paragraph did not make sense. During discussion at the August 13 meeting, IT indicated that they would need to check with their QA/QC staff to understand what was intended.

13. Page 6-1, section 6.1.1: Indicate samples will be identified by marking on the sample container instead of a label.

14. Page 9-1, section 9.0: Include the scheme for data reduction (equations, reporting units), the criteria used to validate data integrity, and methods used to identify and treat outliers in this section.

15. Page 10-3, section 10.2: Include a list of all QC methods that are to be used in the field and in the laboratory.

16. Page 12-1, section 12.0: Include the schedule of maintenance for the laboratory instrumentation.

17. Page 15-1, section 15.0: Include in the text a schedule of reports to management (monthly, quarterly) concerning the assessment of data accuracy, precision and completeness. These reports should also detail audit results and identified QA problems with recommended solutions.

18. Appendix B, no. 10-001-01, section 5.1.2: VOA analysis of water, when unpreserved (no HCl), must be completed within five days VTSR (laboratory data validation - functional guidelines for evaluating organic and inorganic analysis, page 5.)

19. Appendix B, inorganic glassware cleaning, SOP NO. 01-007-00, section 5.2, glassware cleaning. The glassware must be rinsed with a 10% solution of HCl between rinsing the soap off with tap water and the final rinse with deionized water.

Ohio EPA Comments - Volume 4, Health and Safety Plan (HSP) Phase I Task 4 Field Investigation - (August 6, 1990)

1. Ohio EPA's comments regarding site access which were contained in correspondence dated June 15 and July 27, 1990 have not been incorporated into the revised HSP. Revise the first sentence of the first paragraph on page 3-4 to read as follows: "All facility employees and subcontractors who may be ...etc" (delete the phrase "and regulatory"). Revise the first sentence of the second paragraph on this page as follows: "With the exception of regulatory personnel, visitors will not be allowed to enter....(etc.). Add the following sentence to the end of this paragraph: "Regulatory personnel are responsible for providing their own safety equipment and obtaining their own medical monitoring, health and safety training, and respirator fit testing."

2. As indicated in Ohio EPA's correspondence of July 27, 1990, a site map delineating work zones must be provided as required by 29 CFR 1910.120 (d) (3).

Ohio EPA Comments - Volume 3, Appendix A, Draft Standard Operating Procedures and Amendments - (August 13, 1990)

1. In performing the Phase I Task 4 field work, WPAFB is relying on portions of the Draft Field Standard Operating

Procedures submitted by the Air Force July 31, 1990 as Volume 2 Appendix C of the RI/FS Workplan. In order to avoid any delays in the schedule for the Phase I Task 4 Field Investigation, Ohio EPA quickly reviewed the RI/FS Field SOPs that had application to the Phase I Task 4 Field Investigation and provided comments to you in correspondence dated August 2, 1990. Given the tight time frame for the Phase I Task 4 Field Investigation and the condition of the RI/FS Field SOPs as submitted, it was agreed that IT would not spend valuable Phase I Task 4 project time correcting the RI/FS Field SOPs. Instead, IT has been provided with a copy of Ohio EPA's comments on the SOPs and will incorporate the comments into the field work as performed

Proposed Deviations to RI/FS Field SOPs

2. Ohio EPA agreed to consider deviations to the RI/FS Field SOPs that were technically justifiable given the objectives of the Phase I Task 4 investigation. A contractor's differing corporate policy is not considered to be sufficient technical justification for deviating from procedures contained in applicable guidance or procedures worked out between Ohio EPA and WPAFB during the RI/FS Workplan comment resolution meetings. Amendments not acceptable to Ohio EPA or acceptable only with the indicated modifications are identified below. Amendments which are not identified below are acceptable to Ohio EPA for use during the Phase I Task 4 Field Investigation.

3. FP 1-2, Amendment 1: This amendment is not acceptable. If IT wishes to transfer the information from the field notebooks to their own sheets for their own internal use, that is their business. However, the original field information is to be recorded in field logbooks following the procedures identified in the SOP. The field logbooks are to be on-site and available for Ohio EPA inspection during the conduct of the field work.

4. FP 3-1, Amendment 3: This amendment is acceptable if a daily field rinsate blank is taken from the pump following decontamination.

5. FP 5-1, Amendments 4, 5, 8 and 9: These amendments are not acceptable (see FP 1-2, Amendment 1 above).

6. FP 5-2, Amendment 7: This amendment is not acceptable. The grout used to telescope wells should be allowed to cure for a minimum of 24 hours before proceeding.

7. FP 5-4, Amendments 2 and 3: These amendments are not acceptable. If the turbidity requirements are not met after 8 hours of development, development is to stop. Samples for silt and clay analysis and for TOC analysis as necessary will still need to be collected and submitted to the laboratory. The results will be needed to evaluate the remaining data obtained from the affected well.

8. FP 5-5, Amendments 1 and 2: These amendments are not acceptable (see FP 1-2, Amendment 1 above).

9. FP 5-6, Amendment 1: This amendment is not acceptable (see FP 1-2, Amendment 1 above).

10. FP 5-7, Amendment 1: This amendment is not acceptable.

11. FP 6-5, Amendment 3: This amendment is acceptable with the provision that a clear bailer is to be used when checking for free product.

12. FP 7-3, Amendment 2: This amendment is not acceptable (see FP 1-2, Amendment 1 above).

Please note that Ohio EPA comments 12 and 14 through 16 concerning "Volume 3, Sampling and Analysis Plan (SAP), Phase I Task 4" require that additional information be submitted by the Air Force in order to complete the Workplan. Please provide this information concurrent with the written confirmation required by Ohio EPA in condition number 4 above. If you have any questions regarding this letter or if I can be of any further assistance, please do not hesitate to contact me at 285-6357.

Sincerely,

Bonnie D. Bowker

Bonnie D. Bowker
Division of Emergency and Remedial Response

cc: Tom Winston, District Chief/SWDO
Kathy Davidson, TPSU/DERR/CO
Catherine Stroop, Legal/CO
Kathi Nickel, DGW/SWDO
Jack Van Kley, AGO
Wm. Turpin Ballard, USEPA Region 5
Steve Coyle, 2750 ABW/EM

Casing Recommended for Use Where Hard Driving or Corrosive Waters May Be Encountered

(Known as Drive Pipe, Driven Well Pipe, Standard Pipe, Line Pipe, or Reamed and Drifted Pipe)

Nominal Size in Inches	Outside Diameter in Inches	Wall Thickness in Inches	Weight of Plain End Casing in lbs/ft
1.000	1.315	0.133	1.68
1.250	1.660	0.140	2.27
1.500	1.900	0.145	2.72
2.000	2.375	0.154	3.65
2.500	2.875	0.203	5.79
3.000	3.500	0.216	7.58
3.500	4.000	0.226	9.11
4.000	4.500	0.237	10.79
5.000	5.563	0.258	14.62
6.000	6.625	0.280	18.97
8.000	8.625	0.277	24.70
10.000	10.750	0.279	31.20
12.000	12.750	0.330	43.77
14.000	14.000	0.375	54.57
16.000	16.000	0.375	62.58

Casing Suitable for Driving Where Conditions Are Favorable

(Known as Water Well Casing)

Nominal Size in Inches	Outside Diameter in Inches	Wall Thickness in Inches	Weight of Plain End Casing in lbs/ft
3.500	3.500	0.125	4.51
4.000	4.00	0.134	5.53
4.500	4.50	0.142	6.61
5.500	5.50	0.142	8.13
6.000	6.00	0.142	8.88
6.625	6.625	0.156	10.78
8.625	8.625	0.188	16.94

Casing of a nominal size not listed in the above table shall have a thickness not less than that required for the next smaller nominal size listed.

From water well standards

OAC 3745-9-06

INTERNATIONAL
TECHNOLOGY
CORPORATION

August 27, 1990

Mr. Dennis Reed
Battelle Management Operations
Wright Point 2
5100 Springfield Pike, Suite 210
Dayton, Ohio 45431-1231

Re: Response to OEPA Comment Letter of August 20, 1990
PN 199814.03.02

Dear Mr. Reed:

Per your request I have prepared responses to two questions contained in the OEPA letter of August 20, 1990.

Volume 2, Pages 4-13, 4.3.3: Indicate that monitoring wells along the Springfield Street boundary will be sampled at a time when the adjacent WPAFB water supply production wells are not operating. A waiting period after shutdown should be established, which is sufficient to allow flow conditions in the area to return to prepumping conditions prior to sampling the monitoring wells.

Response: Generally, IT has already agreed to shutdown the production wells for some period of time prior to sampling the new wells along Springfield Pike. The issue of concern and confusion with the OEPA comment is the phrase "shutdown should be established which is sufficient to allow flow conditions in the area to return to prepumping conditions."

Initially, we believe that the sampling of the new wells should be reflective of normal Base operations. Normal operations means that the wells along Springfield Pike are cyclically pumped with an average daily withdrawal from the well field at about one million gallons per day.

The West Well Field has been operating for several decades. There can be no return to "prepumping" conditions since the Dayton Rohrer's Island well field has been greatly expanded since the Base wells were installed.

It appears that OEPA is most concerned about what happens when the wells are off for a few hours or days. A particle of water and entrained dissolved contaminants move through the ground-water system at rates less than 8 feet per day. The Base production wells are tens of feet from the Base boundaries, thus the wells would have to be shut off for an extended period before contaminants at the production wells would reach the monitoring wells in Base boundary.

Since prepumping conditions cannot be re-established and since ground-water flow times from the production wells to the monitoring wells are very long, IT will shut the wells down for 24 hours only. This is anticipated to be sufficient time to allow the hydraulic influence of the production wells to be

minimized, but there will be little, if any, affect on the quality of water at the monitoring wells.

Volume 3, Appendix \, FP5-2, Amendment 7: This amendment is not acceptable. The grout used to telescope wells should be allowed to cure a minimum of 24 hours before proceeding.

Response: OEPA provides no rationale for its comment on curing time.

To assist OEPA's evaluation of this comment, the following generalized well construction details are provided:

- 8-inch or 10-inch casing will be driven 2 to 3 feet into the clay confining layer during drilling.

- 6-inch secondary casing will be driven 3 to 5 feet into the clay confining layer.

- Grout will be tremied between 8-inch and 6-inch casings as 8-inch casing is pulled.

- Grout will be allowed to cure for 12 hours (there will be no grout inside the 6-inch secondary casing.

- A 4-inch casing will be driven during drilling through the confining layer. There should be minimal vibration of the 6-inch casing during drilling with the 4-inch casing.

- Following installation of the monitoring well, grout will be tremied into the 4-inch borehole and extend back to land surface.

I hope this information will be helpful to you.

Sincerely,

IT CORPORATION

Bill

William E. Thompson
Project Manager

WET:sdw

Insatallation Restoration Program (IRP)
Environmental Investigation of Ground-Water Contamination
at Wright-Patterson Air Force Base, Ohio

Response to Ohio EPA's comments 12 and 14 through 16 on the "Volume 3, Sampling and Analysis Plan", dated August 20, 1990

12. Page 5-23, second paragraph. This paragraph should be deleted as this information is presented earlier in the section.

14. Page 9-1, section 9.0. Equations and reporting units are presented after each analysis discussion in Section 8.

15. Page 10-3, section 10.2. QC methods used in the laboratory are addressed in Section 13.0 and QC methods used in the field are addressed in Section 5.4.7.

16. Page 12-1, section 12.0. ITAS Laboratory SOPs which contain the schedule of maintenance for applicable laboratory instrumentation are attached.

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-025-00 Date Initiated: 6/23/89 Page 1 of 5 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance In The Metals Laboratory	2452

Prepared by	Approved by	Date	QA Concurrence	Date
Jeff Armie	Ken Mueller	6/20/89	[signature]	7/20/89
			[signature]	6/20/89
			[signature]	7/21/89

1.0 PURPOSE

To help keep an instrument running properly, routine preventive maintenance must be performed. Maintaining an effective routine preventive maintenance schedule requires proper documentation. The purpose of this SOP is to assist the analyst in keeping an effective, well documented routine preventive maintenance log book.

2.0 APPLICATION

This SOP was written specifically for the Perkin-Elmer Plasma II, 3030B, and 560 Atomic Spectrometers. The scope could be modified for additional instruments used in the metals analyses as they added to the laboratory.

3.0 REFERENCES

3.1 Instructional Manuals for the Plasma II Emission Spectrometer, Book 1, 2, and 3.

3.2 Instructional Manual for the 3030B Atomic Absorption Spectrophotometer.

3.3 Instructional Manual for the 560 Atomic Absorption Spectrophotometer.

4.0 ASSOCIATED SOP's

None.

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Plasma II

Maintenance	Check frequency	Replace frequency	Clean frequency
Pump tubing	Daily	Every 2 days	When needed
Capillary tubing	Daily	When needed	When needed
Quartz tubing	Daily	When needed	When needed
Injector tube	Daily	When needed	When needed
Nebulizer insert	When needed	When needed	When needed
RF coil	Daily	When needed	When needed
Quartz bonnet	Daily	When needed	When needed
Drain bottle	Daily		When needed
Purge gas	Daily	When needed	When needed
Purge windows	Daily	When needed	When needed
Vacuum pump oil	Daily	Every 6 mos.	
Adsorption trap		Every month	
Exhaust filter		Annually	
Nitrogen filter		Annually	

5.1.2 3030B

Maintenance	Check frequency	Replace frequency	Clean frequency
Contact rings	Daily	Every 6 mos.	Daily
Graphite tube	Daily	When needed	
Quartz windows	Daily	When needed	When needed
Injector tubing	Daily	When needed	When needed

5.1.3 560

Maintenance	Check frequency	Replace frequency	Clean frequency
Quartz tube assembly	Daily		When needed
Quartz windows	Daily	When needed	When needed

5.2 Documentation in Logbook

Each day, before an analyst begins to run an instrument, the routine preventative maintenance check sheet must be filled out (Figure 1).

5.2.1 Always begin by entering the date in the top left corner of the check sheet.

5.2.2 After the daily checks are made the analyst enters his or her initials in the appropriate column in the check sheet.

5.2.3 In the event that an item on the check sheet is replaced or cleaned, the analyst's initials are entered in the appropriate column on the check sheet.

5.2.4 The comments column of the check sheet is to be used for referencing detailed accounts of all uncustomary repairs, replacements, and maintenance to the back side of the check sheet (Figure 2) by entering "on back" in the space provided. The analyst's initials and the date are to be entered immediately after any comments are entered on the back page.

5.2.5 A yearly calendar is kept in the log book to help remind the analyst of monthly, semi-annual, and annual maintenance.

5.2.6 Preventive maintenance documentation is reviewed monthly by the Metals Team Leader.

PREVENTIVE/ROUTINE MAINTENANCE

Date:

ICP	Check	Replace	Clean	Comments
Pump Tubing				
Capillary Tubing				
Quartz Tubing				
Injector Tube				
Nebulizer Insert				
RF Coil				
Quartz Bonnet				
Drain Bottle				
Purge Gas				
Purge Windows				
Vacuum Pump Oil				

AA 3030B	Check	Replace	Clean	Comments
Contact Rings				
Graphite Tube				
Quartz Windows				
Injector Tubing				

AA 560	Check	Replace	Clean	Comments
Quartz Tube Assembly				
Quartz Windows				

Figure 1

Documentation of Maintenance/Repairs Performed:

Figure 2

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-024-00 Date Initiated: 6/23/89 Page 1 of 9 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the Gas Chromatography/Mass Spectrometry Lab	2452

Prepared by	Approved by	Date	QA Concurrence	Date
Dwight R. Hayes Jr.	C. Caldwell	6/24/89	Janet M. Jones Lauren D. Ross Linda Pleiss	7/20/89 6/29/89 7/21/89

1.0 PURPOSE

This procedure is to be followed to prolong proper operation of the analytical instrumentation and to minimize and correct defects before they result in serious damage or failure.

2.0 APPLICATION

These procedures apply to the instruments used in the Gas Chromatography/Mass Spectrometry Lab.

3.0 REFERENCES

It is the author's belief that the manuals from the instrument's manufacturer should be the source of the "How-to's" for performing preventive maintenance, routine maintenance, and repairs on all the instruments within the applicable laboratory. In that effort, a library of the manuals supplied with the instrument is located within the specific laboratory location for the reference of the analysts as well as technical support personnel.

4.0 ASSOCIATED SOPs

None

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 Once a quarter dust off instrument externally and internally or when repairs are made.

5.1.2 Replace septa with new septa as warranted, when the injection port liner is replaced, or after 50 septum punctures.

5.1.3 Replace Injection Port Liner, if applicable, as warranted when column is "clipped".

5.1.4 Cut about a foot off of a capillary column when the peak tailing becomes excessive.

5.1.5 Replace the capillary column when the chromatographic resolution of the column is no longer suitable for the application.

5.1.6 Check zone temperatures via the computer feedback daily or whenever the temperatures have been changed.

5.1.7 Check the operating gases daily for sufficient volume and pressure.

5.1.8 Check the injection port pressure/flow rate and any split flow rates after any connection change on a column or at a minimum of once a month.

5.2 Autosamplers

5.2.1 Replace wash cup septa once a week or when warranted.

5.2.2 Dispose of waste solvent once a week or as warranted.

5.2.3 Perform the internal diagnostics instrument test on the Varian GCs one a month while visually checking the motion of the autosampler for proper action.

5.2.4 Visually check the injection syringe for leaks at the needle connection daily.

5.3 Mass Spectrometers

5.3.1 General

5.3.1.1 Replace/clean all air filters once a quarter.

5.3.1.2 Dust off the instrument externally and internally once a quarter.

5.3.1.3 Check the operation of all cooling fans once a quarter.

5.3.2 Electronics

5.3.2.1 Dust off and reseat all printed circuit boards semi-annually or when any maintenance is performed on that sub-assembly.

5.3.2.2 Reseat all cable connectors whenever any work is performed within the sub-assembly. Reseat all easily accessed cable connectors semi-annually.

5.3.2.3 Readjust RF resonance on the Finnigan MS after each source cleaning or whenever the rear flange is removed from the manifold.

5.3.2.4 Readjust the emission current via the magnet on the Finnigan MS after each source cleaning or whenever the front flange has been removed or the magnet moved.

5.3.2.5 Check the operation of all solenoid valves semi-annually.

5.3.3 Vacuum/Pneumatics System

5.3.3.1 Change the mechanical roughing pump oil semi-annually.

5.3.3.2 Check the oil level in the mechanical roughing pumps and the turbomolecular pumps monthly.

5.3.3.3 Purge the mechanical pumps monthly for about 15 minutes.

5.3.3.4 Replace desiccant in the vent air drier when the blue desiccant has turned to pink in 75% of the drier.

5.3.3.5 Check the condition of the manifold seals each time the flange is removed from the manifold.

5.3.3.6 Check the flexible tubing quarterly or when work is performed on the pumps.

5.3.3.7 Check that the calibration gas valve and introduction system are functional weekly.

5.3.3.8 Leak check pneumatics system if excessive gas consumption is observed.

5.3.3.9 Leak check vacuum system if excessive pressures are observed on the vacuum gauges or if excessive air is present in spectrum.

5.3.4 Data Systems

5.3.4.1 Clean heads on tape units quarterly.

5.3.4.2 Thoroughly clean printers including ribbon transport system semi-annually.

5.3.4.3 Replace printer ribbons as needed, to maintain enough contrast so that no special effort needs to be taken in reproducing copies of the output due to insufficient contrast.

5.3.4.4 Backup your system software at least quarterly, preferably monthly.

5.4 PURGE AND TRAP EQUIPMENT

5.4.1 Leak check all accessible fittings on the LSC-2, ALS, and their connection to the gas chromatograph on a monthly basis.

5.4.2 Methanol flush the LSC-2 and ALS quarterly or when needed as indicated by carryover contamination, plugged or restricted flow, or suspected adsorption problems.

5.4.3 Check flow rates when any line (other than the front panel connections) has been disconnected or connected.

5.5 THERMAL DESORPTION AND CRYOTRAPPING EQUIPMENT

5.5.1 Check temperatures of each zone monthly or more often if stability of the analysis is a problem.

5.5.2 For the Tekmar 4210, use Section 5.4

6.0 DOCUMENTATION

6.1 Each analyst will keep records for the instrument that he is currently stationed at.

6.2 A checklist will be used to record the date and nature of the preventive maintenance performed. (See Figures 1-4).

6.3 Daily/weekly checklists will be stored in the Maintenance Log Book kept with each Mass Spectrometer. Monthly, quarterly, and semi-annually checklists will be stored in a notebook in the possession of the Technical Support Specialist.

6.4 Preventive maintenance documentation will be reviewed monthly by either the Group Leader of the GC/MS Lab or by the Technical Support Specialist.

7.0 DEFINITION

7.1 When a preventive maintenance action is specified to be performed on a daily basis, it is intended to mean each day that the piece of analytical instrumentation is used, and is not required on the days that the instrument is not used.

Weekly and Daily Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Week Beginning: _____

Preventive Maintenance Action	Week	M	T	W	T	F	S	S
Gas Chromatograph								
Check zone temperatures via computer feedback		☐	☐	☐	☐	☐	☐	☐
Check supply gases for sufficient volume & pressure		☐	☐	☐	☐	☐	☐	☐
Autosampler								
Check or replace wash cup septa	☐							
Check or dispose of waste solvent	☐							
Visually check injection syringe for leakage		☐	☐	☐	☐	☐	☐	☐
Mass Spectrometer								
Check calibration gas valve & response level		☐	☐	☐	☐	☐	☐	☐

Record of routine or conditional preventive maintenance	M	T	W	T	F	S	S
Replacement of GC septa							
Replacement of GC injection port liner							
Clipped GC capillary column							
Replaced GC column							
Cleaned MS source & rods, dipped RF & EC							
Replaced desiccant in vent air drier							
MS manifold seals checked							
MS system leaked checked							
Printer ribbon replaced							
Purge & trap equipment flow rates checked							

Notes & Comments:

Figure 1

Monthly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph ☐ Check the gas pressures and flow rates	_____
Autosampler ☐ Perform Varian instrument test while watching autosampler for proper action	_____
Vacuum Pumps ☐ Check the oil level in the mechanical pumps and in the turbomolecular pumps ☐ Purge the mechanical pumps	_____ _____
Purge & Trap Equipment ☐ Leak check all accessible fittings	_____
Thermal Desorption and Cyrotrapping Equipment ☐ Check temperatures of each zone	_____

Notes & Comments:

Gas Chromatograph/Mass Spectrometry Lab

Figure 2

Quarterly Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Gas Chromatograph	
☐ Dust off instrument internally & Externally	_____
Mass Spectrometers	
☐ Dust off instrument internally & Externally	_____
☐ Replace or clean the air filters	_____
☐ Check the cooling fans	_____
Vacuum Pumps	
☐ Check the integrity of the vacuum tubing	_____
Data Systems	
☐ Clean heads on tape units	_____
☐ Backup the operating software	_____
Purge & Trap Equipment	
☐ Methanol flush LSC & ALS	_____

Notes & Comments:

Gas Chromatograph/Mass Spectrometry Lab

Figure 3

Semi-Annual Preventive Maintenance Checklist

Target Date : _____ Instrument : _____

Performed By : _____ Peripherals : _____

Preventive Maintenance Action	Date Performed
Electronics	
☐ Dust off and reseat all printed circuit boards	_____
☐ Reseat all cable connectors	_____
☐ Check the operation of all solenoid valves	_____
Vacuum Pumps	
☐ Change the mechanical roughing pump oil	_____
Data Systems	
☐ Thoroughly clean printer	_____

Notes & Comments:

Figure 4

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-030-00 Date Initiated: 6/28/89 Page 1 of 7 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the GC Laboratory	2332

Prepared by	Approved by	Date	QA Concurrence	Date
Larry Anderson	A K Caldwell	7/3/89	James W. Jones Lauren D. Rees Linda P. Reeves	7/20/89 7/1/89 7/21/89

1.0 PURPOSE

To outline procedures to be followed for the maintenance and upkeep of the gas chromatographs (GC's), the detectors, and the autosamplers.

2.0 SCOPE

This SOP describes the process of periodic maintenance used on the instruments, detectors, and autosamplers to assure proper performance and quality data.

3.0 REFERENCES

3.1 ITAS-Cincinnati Laboratory Quality Assurance Manual.

3.2 Varian 3300/3400 Gas Chromatograph Operator's Manual, Vol. II.

3.3 Instruction Manual for Model P1-52-02 Photoionization Detector.

3.4 The HALL Book, Tracor Instruments.

4.0 ASSOCIATED SOPS

None.

5.0 PROCEDURE

5.1 Gas Chromatographs

5.1.1 The septum inside the GC injection port is to be changed after each project or at a time determined by the analyst so that the calibration of the instrument is within required specifications.

5.1.2 Each column inside a GC should be constantly watched during an analysis for possible deteriorating performance, such as excessive breakdown, high baseline, or non-resolution of target peaks. If such performance is detected, the column should be pulled from the GC, and the head of the column repacked and the glass wool plug replaced. If in the case of pesticide analysis, separation of A-BHC and B-BHC or endrin ketone and DBC doesn't occur, the column packing should be replaced.

5.1.3 Supply gases are checked every day to make sure that flow to the GCs and detectors are at appropriate levels.

5.1.4 Gas line traps are changed when baseline levels and noise are excessive or when it is determined by the analyst that calibration is outside required specifications.

5.2 Detectors

5.2.1 Electron Capture Detectors

Preventative maintenance on the EC detectors is difficult. However, if an ECD is performing poorly here are several suggestions. First try baking the detector out at about 400°C. This is normally all that is necessary to clean out a dirty detector. If that doesn't work try running hydrogen gas through the lines and into the detector for about 10 minutes. Refer to Varian 3300/3400 Operator's Manual, Vol. II.

5.2.2 Flame Ionization Detectors

FIDs collect a lot of soot from the burning process of the detector, so periodically (every 6 months) the FID is taken apart so the flame tip can be cleaned. The flame tip is first cleaned of soot and dirt with an emery board. A wire brush may help. Then sonicate the flame tip with methylene chloride, hexane, and methanol in that order. It is also a good idea to use the emery paper on the electrodes going to the FID tower. Refer to Varian 3300/3400 Operator's Manual, Vol. II.

When reassembling the detector install a new aluminum seal washer onto the shoulder of the detector base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3300/3400 Operator's Manual, Vol. II.

5.2.3 Photoionization Detector

CAUTION: When disassembling the detector, use disposable rubber gloves or tweezers to avoid contamination of internal components.

The PID is cleaned when the baseline level and the noise are excessive and when it is determined by the analyst that calibration is outside the required specifications. To clean the 10.2 eV UV lamp first turn off the lamp and line power. Allow the detector to cool to ambient temperature before handling. Remove the four lamp housing screws from the rim of the detector assembly and carefully remove lamp housing. Lift the light source from detector. Check the light source window for deposits, films, or discoloration by looking at a reflected image on the window. Then use a clean Kimwipe or other non-linting tissue dampened with water to remove

any water soluble film. Next, add a drop of cleaning compound (supplied by HNU) to the window surface. Use a clean Kimwipe and scour the surface in a circular motion to remove film. Be careful not to scratch the window. Rinse the window with an 80°C solution of mild dish-washing liquid in water and rinse finally with 80°C distilled water. Dry the window in air or with lint free tissue before reassembling the detector. To reassemble the PID, position the disc and lamp window seal in place and place the lamp in position over the lamp window seal making sure the seal is properly positioned to insure against leaks. Carefully place the lamp housing over the lamp and insert and tighten the four screws. Once re-assembled, check the flowrate at the PID exhaust. Refer to PID instruction manual.

5.2.4 Thermionic Specific Detector

This detector is also known as the nitrogen-phosphorus detector. When the TSD exhibits low sensitivity or high background noise cleaning is necessary. Visual inspection may also indicate a need for cleaning since the ceramic bead, collector, or flame tip are all parts that are prone to deposits.

Bead Cleaning: deposits can be removed from the bead by gently scraping the bead surface with a sharp tool or abrasive paper. Support the bead while cleaning to prevent bending the lead and cracking the ceramic coating on the leads.

Flame Tip and Internal Parts Cleaning: handle internal parts with tweezers and place on a clean, uncontaminated surface. Clean deposits from the surface of the collector with water or emery paper. Remove deposits which form on top of the flame tip by scraping. If the flame tip is plugged, clear by inserting wire into flame tip orifice. Flush the cleaned components with methanol and air dry.

When reassembling the detector, install a new seal washer onto the shoulder of the detector base. This should be done each time the detector is installed. For best results, tighten the screws to the tower a little at a time so that equal pressure is applied to the seal. Refer to the Varian 3300/3400 Operator's Manual, Vol II.

5.2.5 HALL Electrolytic Conductivity Detector

Preventative maintenance for the HALL detector consists of checking to make sure the solvent in the solvent reservoir is at the correct level. The flow from the solvent reservoir must also be checked periodically. A changing flow rate may indicate a leak or clog in the transfer lines. The flow rate should be about 0.5 ml/min. Refer to the HALL book.

5.3 Autosamplers

5.3.1 Dynatech Precision Autosamplers

The septa (2) are replaced once every six months or after it is determined by the analyst that calibration is outside the required specifications of analysis. Close attention is paid to an autosampler when high levels of targets are passed through it to see that no cross-contamination is taking place. Corrective action is taken if such a situation arises. The first injection made by the autosampler is always watched to see if the injection sequence is working properly.

5.3.2 Varian 8000 Autosampler

The septum is replaced once very three months or after it is determined by the analyst that calibration is outside the required specifications of the analysis.

5.4 The Preventative Maintenance Records

5.4.1 Records of preventative maintenance and repairs must be kept in all situations. For this, a logbook has been designed and placed beside each instrument. If an instrument has two detectors that can be used simultaneously, two logbooks will accompany that instrument.

5.4.2 The check list (Figure 1) is filled out by first designating the instrument ID such as V, or A, or K. Next, the week the check list is kept is recorded. The top portion of the list is a quick reference to the availability of the instrument. If for example, an analyst changes the septum and replaces the gas line trap on the instrument the analyst just initials the box pertaining to the action that day.

5.4.3 The bottom portion of the list is filled out when routine maintenance and repairs are made to the instrument. A detailed description of the action performed on the instrument is written down with the analyst's initials and date(s) when the repair took place.

5.4.4 Another quick reference guide used to keep track of when instruments were repaired or maintenanced is on the scheduling board. A tag is placed on the board under the instrument identifier with a small summary of the repair/maintenance on it. This will allow better tracking of when an instrument was maintenanced or when a detector was last cleaned. If more information is needed about the repair, refer to the preventative maintenance check list by the date on the tag.

5.4.5 Preventative maintenance documentation is reviewed monthly by the Group Leader or Team Leader.

WEEKLY PREVENTATIVE MAINTENANCE CHECK LIST WEEK OF _____

INSTR. I.D. _____

ITEM REQUIRED		FREQUENCY	MONDAY	TUESDAY	WEDNESDAY	THURSDAY	FRIDAY	SATURDAY	SUNDAY
CHANGE SEPTUM	FRONT	A,D,I							
	BACK								
CHANGE A/S SEPTUM		F							
EC(N163) WIPE TEST		B							
CHANGE GAS LINE TRAPS		D,E							
REPLACE COLUMN PACKING		B							
CLEAN DETECTOR		D,E							
CHANGE GLASS WOOL PLUG		D,E							

KEY: A. AFTER EACH PROJECT
B. DETERMINED BY ANALYST SO THAT CALIBRATION IS WITHIN REQUIRED SPECIFICATIONS
C. AFTER 3 INJECTIONS
D. ONCE EVERY THREE YEARS - ECD ONLY
E. WHEN BASE LINE LEVEL AND NOISE ARE EXCESSIVE
F. ONCE EVERY THREE MONTHS
I WHICHEVER IS MORE FREQUENT

DATE	INT.	PREVENTATIVE MAINTENANCE / ROUTINE MAINTENANCE

DATE	INT.	REPAIRS MADE TO INSTRUMENT

Figure 1

INTERNATIONAL TECHNOLOGY CORPORATION	SOP *Standard Operating Procedure* ITAS Cincinnati	SOP No. 01-027-00 Date Initiated: 6/26/89 Page 1 of 13 Date Revised:

Title:	Word Processing Disk Number:
Preventive Maintenance in the Wet Chemistry Laboratory	2332

Prepared by	Approved by	Date	QA Concurrence	Date
Ken Mueller	C.H. Caldwell	6/30/89	Janet M. Lohr Lauren Drees Linda Reeves	7/20/89 7/1/89 7/24/89

1.0 PURPOSE

This SOP describes the maintenance procedures taken on a daily, weekly, or monthly basis to prevent avoidable breakdowns of instruments in the wet chemistry laboratory.

2.0 APPLICATION

This SOP applies to the following instruments:

- Waters Ion Chromatography System
- Technicon TRAACS 800 Auto Analyzer System
- Dohrman/Xertex DC 80 TOC Analyzer
- Dohrman/Xertex DX 10 TOX Analyzer
- Milton Roy Spec 21 UV/Vis spectrophotometer
- Orion Model 720 pH/mv meter
- YSI Model 34 Specific Conductance meter
- Millipore Milli-Q H_2O System

3.0 REFERENCES

3.1 Operator's manual for the Waters model 431 conductivity meter, Model 731 pump, Model 715 Ultra Wisp autosampler.

3.2 Operator's manual for the Technicon TRAACS 800 auto analyzer system.

3.3 Operator's manual for the Dohrman/Xertex TOC analyzer.

3.4 Operator's manual for the Dohrman/Xertex TOX analyzer.

3.5 Operator's manual for the Milton Roy Spec 21 UV/Vis spectrophotometer.

3.6 Operator's manual for the Orion Model 720 pH/mv Meter.

3.7 Operator's manual for the YSI model 34 Specific Conductivity Meter.

3.8 Operator's manual for the Millipore Milli-Q H2O system.

4.0 ASSOCIATED SOPS

None

5.0 PROCEDURE

5.1 Preventive Maintenance Schedules

5.1.1 Waters Ion Chromatography System

Maintenance	Frequency
Filter and Degas Eluent	With each batch of eluent
Check solvent reservoir filter	With each batch of eluent
Check inlet line for bubbles	Daily, when in use
Check needle wash reservoir	Daily, when in use
Clean or replace guard column	As needed
Clean or replace ion column	As needed
Wipe down outside of instrument	As needed

5.1.2 TRAACS Auto Analyzer System

Maintenance	Frequency
Check air pressure (22 psi +/- 2)	Daily, when in use
Remove tubing from air valves	Daily, when in use
Check base and gain settings	Weekly, when in use

(continued)

Maintenance	Frequency
Change pump tubing	Monthly
Replace pump platens	Every 6 months
Replace colorimeter lamp	Yearly
Replenish reagents	As needed
Wipe down outside of instrument	As needed

5.1.3 Dohrman/Xertex DC 80 TOC Analyzer

Maintenance	Frequency
Replace septa	As needed
Replace glass wool in boat	Daily, when in use
Replenish reaction vessel reagent	Daily, when in use
Repack tin and copper scrubbers	As needed
Replace pump tubing	As needed
Repack lithium hydroxide scrubber	As needed
Repack cupric oxide furnace tube	As needed
Wipe down outside of instrument	As needed

5.1.4 Dohrman/Xertex DX 10 TOX Analyzer

Maintenance	Frequency
Check inlet and outlet tubes for residue buildup	Daily, when in use
Clean or replace inlet and outlet tubes	As needed
Check all electrodes for residue buildup	Daily, when in use
Clean or replace cell electrodes	As needed
Replenish electrolyte solution	As needed
Replace glass columns and "O" rings	As needed
Wipe down outside of instrument	As needed

5.1.5 Milton Roy Spec 21 UV/Vis Spectrophotometer

Maintenance	Frequency
Check instrument response with commercially purchased solutions	Monthly
Check wavelength with didymium filter	Monthly
Wipe down outside of instrument	As needed

5.1.6 Orion Model 720 pH/mv Meter

Maintenance	Frequency
Clean electrode	As needed
Wipe down outside of instrument	As needed

5.1.7 YSI Model 34 Specific Conductivity Meter

Maintenance	Frequency
Clean conductivity cell	Quarterly
Replatinize cell	As needed

5.1.8 Milli-Q H_2O System

Maintenance	Frequency
Change deionizing tanks	As needed
Change Milli-Q cartridges	As needed
Conductivity check	Daily

5.2 Documentation of Maintenance

5.2.1 Each time a maintenance check or procedure is performed it is recorded on the appropriate maintenance log sheet (Figures 1-8) and kept in books near each instrument.

5.2.2 When a routine check or procedure is performed, the analyst dates and initials the appropriate line on the log sheet. If a replacement is made, it is noted in the space provided for additional information.

5.2.3 When a non-routine maintenance procedure is performed, a description is entered in the space provided for additional information as well as the date and the name of the person performing the procedure.

5.2.4 All preventive maintenance documentation is reviewed by the lab supervisor monthly.

PREVENTIVE / ROUTINE MAINTENANCE
WATERS ION CHROMATOGRAPHY SYSTEM

Daily Basis

Check Inlet Line for Bubbles & Needle Wash Reservoir

Date	Initials	Date	Initials

Whenever New Eluent is Made

Eluent Filtered & Degassed, & Solvent Reservoir checked

Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Clean guard column		
Replace guard column		
Clean ion column		
Replace ion column		

Checked By:_____ Date:_____

Figure 1

PREVENTIVE / ROUTINE MAINTENANCE
TRAACS 800 AutoAnalyzer System

Daily Basis

Check Air Pressure & Remove Tubing from Air Valves

Date	Initials	Date	Initials

Weekly Basis

Check Base & Gain Settings

Date	Initials	Date	Initials

Monthly Basis

Replace Pump Tubings

Date	Initials

6 Month Basis

Replace Pump Platens

Date	Initials

Yearly Basis

	Date	Initials
Replace Colorimeter Lamp		

Checked By:_____ Date:_____

Figure 2

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DC 80 TOC ANALYZER

Daily Basis

Replace Glass Wool

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Replenish Persulfate Sol'n

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Repack Tin & Copper Scrubbers		
Replace pump tubing		
Repack Lithium Hydroxide Scrubber		
Repack Cupric Oxide tube		

Checked By:_____ Date:_____

Figure 3

PREVENTIVE / ROUTINE MAINTENANCE
DOHRMANN DX 20 TOX ANALYZER

Daily Basis

Check Inlet & Outlet Tubes For Residue Buildup

Date	Initials	Date	Initials	Date	Initials

Daily Basis

Check Cell Electrodes For Residue Buildup & Replenish Electrolyte

Date	Initials	Date	Initials	Date	Initials

As Needed Basis

	Date	Initials
Clean Inlet & Outlet Tubes		
Replace Inlet & Outlet Tubes		
Clean Cell Electrodes		
Replace Cell Electrodes		

Checked By:_____ Date:_____

Figure 4

PREVENTIVE / ROUTINE MAINTENANCE
MILTON ROY SPECTRONIC 21 UV/VIS SPECTROPHOTOMETER.

Monthly Basis

Check Response with Commercially Prepared Solutions

Date	Initials	Date	Initials

Monthly Basis

Check Wavelength with Didymium filter

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 5

PREVENTIVE / ROUTINE MAINTENANCE
pH METER & ELECTRODE

Monthly Basis

Clean Electrode & Meter

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 6

PREVENTIVE / ROUTINE MAINTENANCE
CONDUCTIVITY METER

Quarterly Basis

Clean Cell & Meter

Date	Initials	Date	Initials
_____	_____	_____	_____
_____	_____	_____	_____

As Needed Basis

Re-Platinize Cell

Date	Initials	Date	Initials
_____	_____	_____	_____
_____	_____	_____	_____

Additional Maintainence Documentation

_____
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Checked By:_____ **Date:**_____

Figure 7

PREVENTIVE / ROUTINE MAINTENANCE
D.I. H2O QUALITY SYSTEM

Change D.I. Tanks

As Needed Basis

Date	Initials	Date	Initials

Change Milli-Q Cartridges

As Needed Basis

Date	Initials	Date	Initials

Additional Maintainence Documentation

Checked By:_____ Date:_____

Figure 8

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